AFML-TR-68-347
PART I

THERMAL DEGRADATION OF POLYAMIDES

PART I. ALIPHATIC POLYMERS

I. J. GOLDFARB

A. C. MEEKS

TECHNICAL REPORT AFML-TR-68-347, PART I

FEBRUARY 1969

AIR FORCE MATERIALS LABORATORY
AIR FORCE SYSTEMS COMMAND
WRIGHT-PATTERSON AIR FORCE BASE, OHIO

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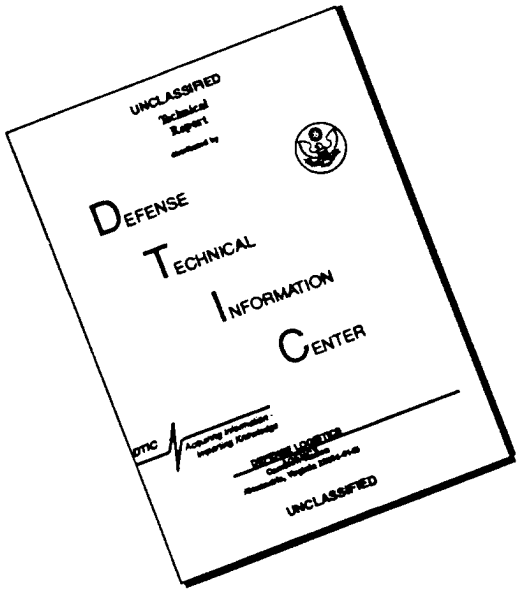

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FOREWORD

This report was prepared by the Polymer Branch, Nonmetallic Materials Division, Air Force Materials Laboratory. The work was initiated under Project No. 7342, "Fundamental Research on Macromolecular Materials and Lubrication Phenomena," Task No. 734203, "Fundamental Principles Determining the Behavior of Macromolecules." It was administered under the direction of the Air Force Materials Laboratory, Wright-Patterson Air Force Base, Ohio, with Dr. I. J. Goldfarb (MANP), Task Scientist.

The report covers work conducted from September 1966 to June 1968. It was submitted by the authors in November 1968.

The authors wish to thank Mr. W. Baltzell and Mr. R. R. Luthman, Jr., for their valuable assistance in the experimental work and in the calculations. Thanks are also due Dr. H. Friedman and Dr. H. Goldstein, General Electric Space Sciences Laboratory for the mass spectral analysis.

This technical report has been reviewed and is approved.

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ABSTRACT

The thermal degradation of two aliphatic polyamides, polyhexamethylene adipamide (nylon 6.6) and polyhexamethylene sebacamide (nylon 6.10) have been studied. Molecular weight changes, weight loss, and volatile product analysis were used to help elucidate the reaction mechanisms.

The presence of low molecular weight material and polymerizable end groups in these polymers complicated the interpretation of molecular weight changes during degradation. The weight loss data obtained allowed the calculation of rate data. Nylon 6.6 degradation gave an activation energy of 45 kcal/mole while nylon 6.10 degradation was characterized by an activation energy of 55 kcal/mole. Both polymers gave evidence of random scission kinetics. The volatile products were consistent with the occurrence of further condensation, scission, and cross-linking reactions.

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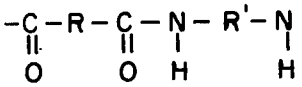
SECTION I

INTRODUCTION

A wealth of information is available in the literature concerning the degradation of vinyl polymers (References 1 and 2) and in some cases complete analyses of mechanism are well established. However, there seems to be a deficit of similar information for polycondensates except perhaps for some polyesters (e.g., polyethylene terephthalate). In view of the considerable current interest in polymers of this type, this is somewhat surprising, especially since the more exotic polycondensates (e.g., polybenzimidazoles, poly(bis-benzimidazobenzophenanthroline) have shown considerable promise as thermally stable materials of improved useful service life.

A large number of polyamides containing the repeating unit



are available where R and R' range from short aliphatic hydrocarbon chains to aromatic and heterocyclic rings. Many of these polymers have useful physical and chemical properties and have achieved commercial importance as textiles and molding compounds.

One source of such a diversified range of structures has been the recent interest in increasing the useful life of polymeric materials at high temperature, particularly by the incorporation of aromatic rings into the backbone of the polymer. Russian research on polyamides has been particularly active (References 3 and 4) as has the work being carried out at Chemstrand Research Center (References 5 and 6).

Information is available on the composition of the evolved gases during degradation (Reference 7) but there has been relatively little interest in the rates of the various processes or the thermodynamic parameters which control the degradation of these compounds.

The work described here has been an attempt to correlate polyamide structure with the mechanism and kinetics of the degradation reactions. Early studies were devoted to molecular weight changes which were expected to take place at temperatures below those required for the onset of drastic weight loss of the polymer. Temperatures not far above the sample melting point were used but 1 to 2% weight loss was often evident. In this way, it was hoped to be able to follow molecular weight changes as a function of the exposure time, temperature, and polymer structure. Since undesirable changes in physical properties often accompany molecular weight changes, knowledge of the kinetics which govern molecular weight changes could be of use in predicting polymer lifetimes under various conditions. The main objective here, however, was a determination of the types of reaction (scission, etc.) responsible for molecular weight degradation.

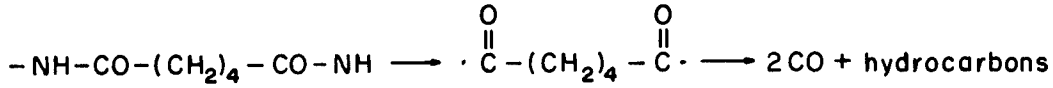
The studies were extended to follow the kinetic laws involved in weight loss processes. This included evaluation of methods employed for the determination of kinetic parameters involved in weight loss processes (Reference 8). Measurements were made under both isothermal and linearly increasing temperature conditions, and machine methods for calculation of the results were devised (References 8 and 9). Some of the results of this work have been reported previously (Reference 10), but since a better method has been devised for the calculations they are repeated here.

This report is concerned with the weight loss, molecular weight changes, production of volatiles, etc., of poly(hexamethylene adipamide) and poly(hexamethylene sebacamide) designated as nylon 6.6 and nylon 6.10, respectively. The numbers represent, in order, the number of carbon atoms in the diamine and the diacid constituents of the polymer chain.

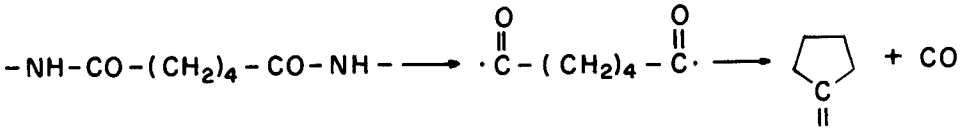
SECTION II
PREVIOUS INVESTIGATIONS

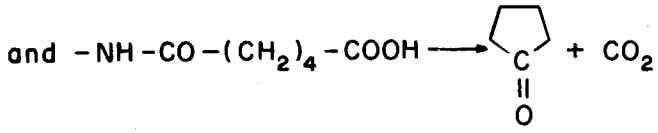
In several of the early investigations into the degradation of polyamides (Reference 11), copolymers, e.g., of nylon 6.6 and nylon 6.10, were used. The use of such materials complicates the interpretation of the results of degradation particularly if the possibility of the formation of new structures by transamidation exists.

Achhammer, et al. (Reference 12) described a considerable amount of information on the degradation of a series of copolyamides. Changes in mechanical, electrical, and other properties were measured as a function of the time of exposure to artificial weather, etc. The gaseous products detected during exposure to high temperatures were water, carbon dioxide, carbon monoxide, hydrocarbons and ethanol (solvent). The source of water was suggested to be a cross-linking reaction and it was proposed that carbon monoxide and hydrocarbons were evolved during a series of scission reactions:



A significant quantity of cyclopentanone was detected in the pyrolysis gases, a possible mechanism for its formation being





Sufficient CO and CO_2 were present in the gaseous products to account for the formation of cyclopentanone by both of these mechanisms. When the polymer contained sebacic acid units, no CO was evolved and no cyclic hydrocarbons were detected. The 10 carbon cyclic ketone would not be stable under degradation conditions.

The quantity of CO_2 produced was 10 times in excess of that expected on the basis of end groups alone, showing its source to be either absorbed CO_2 or that produced by some unknown mechanism from parts of the polymer chain other than end groups.

Goodman (Reference 13) investigated the decomposition products of a series of N,N'-di-n-butylamides as model compounds for nylon polymers. Dibutyladipamide decomposed almost completely giving n-butylamine, minor amounts of CO and hydrocarbons and substantial quantities of CO_2. No cyclic ketone was detected in conflict with expectations based on Achhammer's work (Reference 12). Goodman claimed to have established a unique reaction of N-substituted adipamides in which CO_2 is produced on heating without the formation of equivalent quantities of hydrocarbons. The composition of the residue was examined and shown to contain nitrogen. It was postulated that the nitrogen was present in a 5-membered heterocyclic ring. It was later shown (Reference 14) that residues from the degradation of both dibutyladipamide and nylon 6.6 contained 5- and 6-membered rings as well as a pyrrole derivative.

Kammerbeek, Kroes, and Grolle (Reference 15) published a considerable body of information on the gellation and the thermal degradation of nylon 6 and nylon 6.6 and postulated a series of possible reactions to account for the cross-linking and for the composition of the gaseous products. Some information on the changes in molecular weight during heat treatment were also presented. To back up the postulated mechanisms, authentic specimens of the residue structures were prepared and examined.

For nylon 6, the reactions suggested by these authors are given below.

(A) Primary Reaction

Scission of the bond in the β position to the carbonyl group.

$$-CO-NH-(CH_2)_4-CH_2-CO-NH-CH_2-(CH_2)_4-CO-NH \cdots I$$

$$I \longrightarrow -CO-NH-CH_2-(CH_2)_4-CO-NH_2 \cdots II$$

$$+CH_2 = CH-CH_2-CH_2-CH_2-CO-NH-$$

The amide (II) may then split off water to leave the nitrile.

Also scission of the -NH-CH_2- bond may occur:

$$I \longrightarrow -CO-NH-(CH_2)_5-CO-\overline{N}I + CH_3-(CH_2)_4-CO-NH-$$

$$\downarrow \text{ Rearranges}$$

$$-CO-NH-(CH_2)_5-N=C=O$$

(B) Secondary Reactions

Chain end hydrolysis

$$R-NH-CO-R' + H_2O \longrightarrow RNH_2 + R'COOH$$

Condensation

$$R-COOH + HOOC-R' \longrightarrow R-CO-R' + H_2O + CO_2$$

$$R-NH_2 + R'-NH_2 \longrightarrow R-NH-R' + NH_3$$

Several other processes which modify the chain were also proposed.

Straus and Wall (Reference 11) examined the effects of the deliberate addition of impurities, e.g., phosphoric acid, to nylon 6. A threefold increase in the maximum rate of weight loss was observed with this acid, probably because of the greater importance of ionic processes. The same authors (Reference 16) also showed that purification of the polymer decreased the rates of degradation and of production of CO_2 and increased the activation energy of the overall weight loss. Their sample of nylon 6 was extensively purified by extraction with solvents and dried well since it was claimed that the presence of moisture could cause hydrolytic decomposition, with the production of CO_2, which overshadows the normal free radical thermal decomposition. However, it was shown that the quantity of CO_2 evolved could never be reduced to that expected on the basis of end groups alone and it was suggested that even after being dried carefully the polymer still contained absorbed water capable of causing hydrolysis of the polymer. Cyclopentanone was detected in the gaseous products the quantity of which decreased after the polymer was treated with acid. Thus the production of the cyclic ketone is essentially a free radical process but not occurring at adipic end units.

The authors determined an activation energy of 43 kcal/mole for the weight loss of nylon 6.6 from the maximum rates of weight loss but suggested the true value for the pure free radical reaction with no hydrolysis component should be between 50 and 60 kcal/mole.

An early attempt at the determination of kinetic parameters for the pyrolysis of polyamides was made by Straus and Wall (Reference 11) using nylon 6 and mixtures of copolyamides. A wide range of activation energies (14 to 42 kcal/mole) was determined from isothermal weight losses but their highest value was considered to be more representative of the pure, free radical decomposition.

SECTION III

EXPERIMENTAL

1. PREPARATION OF POLYMERS

Nylon 6.6 and nylon 6.10 were received from Chemstrand Research Center in the form of fiber and chopped ribbon. Both materials were prepared from high purity intermediates without the addition of stabilizers or other additives (Reference 17). Both the polymers were reduced to a finely divided form by precipitation with water from formic acid solution following the procedure described in Reference 10. Traces of unreacted material and low molecular weight polymer were removed by extraction with hot water for a minimum of 8 hours.

2. INTRINSIC VISCOSITY MEASUREMENTS

All measurements were made using standard or Semi-Micro Cannon-Ubbelohde dilution viscometers. Formic acid solutions required a number 75 viscometer and a number 150 or 200 was used for m-cresol solutions.

3. END GROUP TITRATIONS

End group titrations were carried out on m-cresol solutions of the polymer using 0.01N alcoholic HCl and NaOH solutions. The polymer solution was contained in a small cell through which nitrogen could be passed to prevent oxidation of the solvent. The end point was determined by the inflection in the curve of conductivity versus volume of titrant added. Conductivity was measured using platinum electrodes and the Thomas SERFASS conductivity bridge Model RCM 15B1.

4. VAPOR PRESSURE OSMOMETRY (VPO)

Measurements of number average molecular weight were made using the Mechrolab Vapor Pressure Osmometer Model 302. The auxilliary high temperature chamber was used at 130°C for a limited number of measurements but normally 37°C and 65°C were used when fluoroalcohols were employed as

solvents. Early in this work, difficulty was encountered in obtaining reproducible results and the cause was traced to variations of sample drop size, a previously unreported phenomenon. References 18 and 19 describe in detail the techniques used for correction of time and drop size effects and Reference 20 describes the modification made to the instrument to permit recorder plotting of the VPO output.

5. WEIGHT LOSS MEASUREMENTS

Ainsworth Thermobalances, Models AV and RV, were used throughout this work, the former giving a full scale recorder pen deflection equivalent to a 100 mg weight change, the latter to a 10 mg change. A complete description of the apparatus and experimental technique is given in Reference 10.

SECTION IV

CHARACTERIZATION AND MOLECULAR WEIGHT DETERMINATION OF POLYAMIDES

1. SOLUBILITY

The literature is replete with descriptions of the determination of the molecular weight of polyamides, in most cases nylon 6.6. One of the major difficulties is the choice of a solvent suitable for the particular technique being employed.

Formic acid solution (85-97%) readily dissolves aliphatic polyamides but protonation of the NH group causes complications in the determination of viscosity (References 21 and 22). Meta cresol is a useful solvent but the solutions readily become colored by oxidation of the m-cresol. Purification of nylon by precipitation from m-cresol is not recommended since the color contaminates the precipitate.

Stronger acids, trifluoroacetic, sulfuric and methane-sulfonic, readily dissolve aromatic polyamides but the possibility exists for hydrolysis of aliphatic materials.

It has been reported (Reference 23) that a saturated solution of calcium chloride in methanol will dissolve some polyamides but nylon 6.10 is not included.

Recently several fluorinated alcohols have become readily available; they are especially useful in the determination of molecular weight by VPO (Reference 24) because of their compatibility with the materials of construction of the VPO and they have been used in the measurement of other solution properties (References 25 and 26). Nylon 6.10 dissolves readily in 2,2,2-trifluoroethanol on warming but at room temperature some solutions tend to be unstable (see Section IV.4).

No single solvent could be employed in this work since several different measuring techniques were used each having certain unique solvent requirements.

2. VISCOMETRY

The intrinsic viscosities (I.V.) of nylon 6.10 in m-cresol and in 85% formic acid have been measured using various samples of polymer. Identical I.V. values were obtained using several batches of purified polymer but material which had not received the water extraction process had a distinctly lower I.V. than extracted polymer. A composite of the viscosities of the extracted material is shown in Figure 1.

In formic acid, at concentrations between 0.2 and 0.8 g/dl, the data may be represented by a straight line which gives an intrinsic viscosity of 0.74 dl/g. It is well known that polyamides exhibit polyelectrolyte effect manifested by anomalously high values of η_{sp}/c at low concentrations (References 21 and 22) but this was not observed in the concentration range used here.

In m-cresol anomalously low values of η_{sp}/c were observed at concentrations below 0.1 g/dl. The intrinsic viscosity determined from the linear part of the curve is 1.15 dl/g. Viscosity data for nylon 6.6 (Reference 27) shows

$[\eta]$ formic acid/$[\eta]$ m-cresol = 0.9, but we find the ratio to be 0.64 for nylon 6.10.

In order to convert I.V. into viscosity average molecular weight, the constants K and α on the Mark Houwink equation $[\eta] = KM^{\alpha}$ are necessary. These apparently have not been determined for nylon 6.10 but data is available for nylon 6.6 (References 28 and 29). For nylon 6.6 in 90% formic acid (Reference 29), $K = 11 \times 10^{-4}$ and $\alpha = 0.72$ in the molecular weight range between 5000 and 25,000. The value of K for nylon 6.10 should be larger because of the greater molecular size. Using these constants for the nylon 6.10 data gives an estimate for $\overline{M}_v$ of 8500.

For a mixed polyamide in m-cresol (Reference 30), $K = 0.29 \times 10^{-6}$ and $\alpha = 1.3$. If $[\eta] = 1.15$ then $\overline{M}_v = 1.2 \times 10^5$.

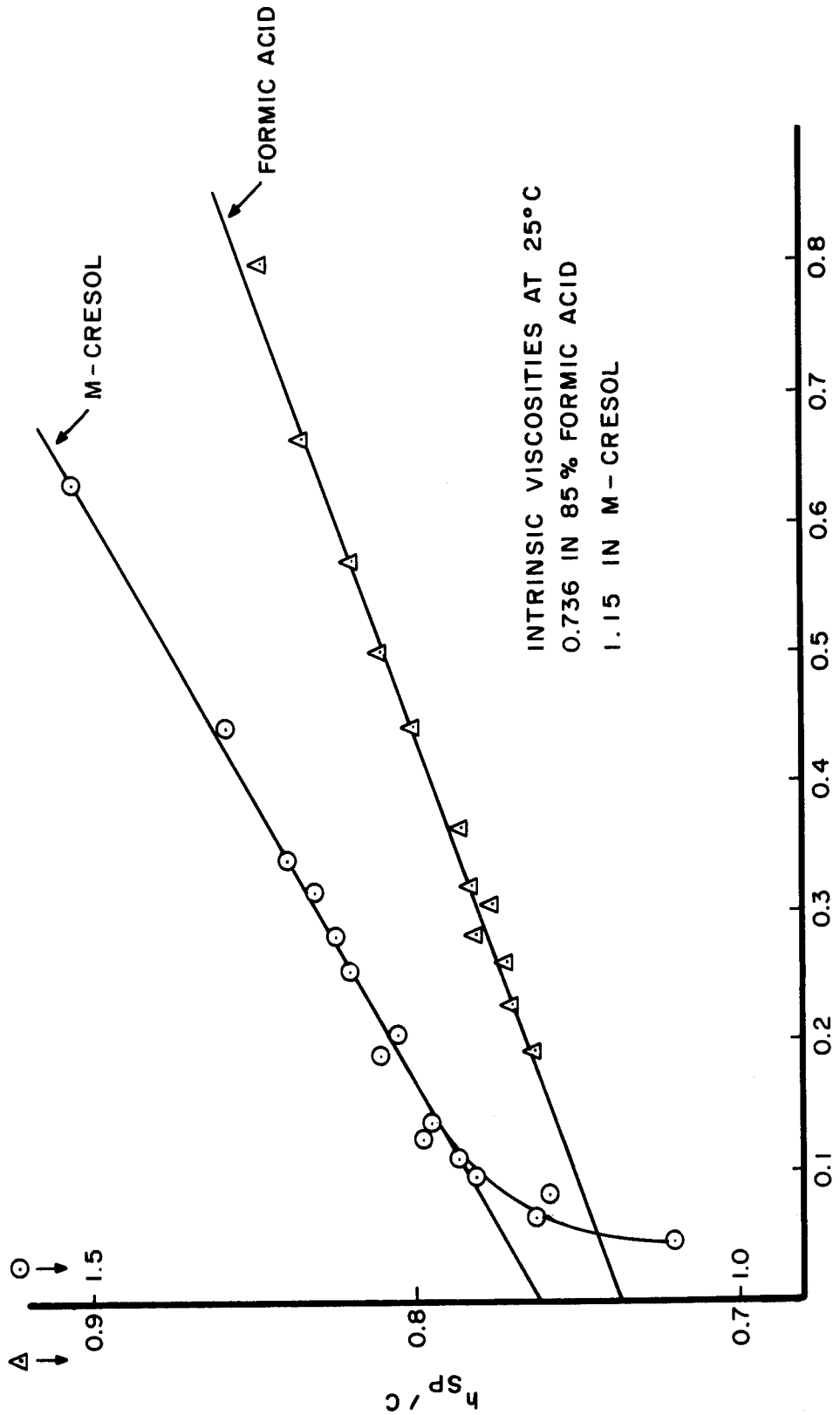


Figure 1. Intrinsic Viscosity Plots for Nylon 6.10

Ohama and Ozawa (Reference 24) have quoted figures for several nylons: Nylon 6, $\overline{M}_v = 10,400\left[\eta\right]^{1.61}$; Nylon 7, $\overline{M}_v = 17,000\left[\eta\right]^{1.4}$ and Nylon 9, $\overline{M}_v = 18,600\left[\eta\right]^{1.4}$ in m-cresol at 25°C. It has been shown that K and α are the same for nylon 6.6 and nylon 6 (Reference 31), so the constants seem to depend mainly on the number of carbon atoms in the chain. If this is so, the constants for nylon 6.10 might be similar to those for nylon 8. Using the relationship $M = 18,000\left[\eta\right]^{1.4}$ interpolated from the nylon 6, 7, and 9 data, $\overline{M}_v = 21,900$ for the nylon 6.10 used here.

The nylon 6.6 used here has an intrinsic viscosity of 0.63 dl/g in m-cresol at 25°C. The molecular weight $\overline{M}_v$ varies depending on which literature values of K and α are used.

Thus, it can be seen that no reliable estimate of the molecular weights of the starting polymers could be arrived at using published K and α data. Measurements of I.V. should still be a reliable indication of changes in molecular weight if it can be assumed that the reaction causing the molecular weight change does not alter the residue structure (e.g., degree of cross-linking) significantly. This implies that the constants K and α apply to both the starting material and the degraded polymer.

To test the effect of the purification procedure on the thermal behavior of nylon 6.10, several series of degradations were carried out at 289°C for varying times and intrinsic viscosities were measured. The results, plotted in Figure 2 show that considerable variations in I.V. of the degraded material are caused by batch changes. All batches were derived from the same original material.

Batches B and C had both been freeze dried and extracted with water in a Soxhlet but had slightly differing I.V.'s (1.15 and 1.17, respectively). Batch E was similarly prepared but was not extracted to remove low molecular weight materials. The I.V. of this batch was 0.92 which reflects the presence of low molecular weight components. The amount of material extracted during the Soxhlet treatment was only 1.3%. The thermal behavior of Batch E is in keeping with this. There is a rapid rise in I.V., probably due to continuation of polymerization but after 200 minutes exposure at 289°C, the I.V. falls as the importance of scission increases.

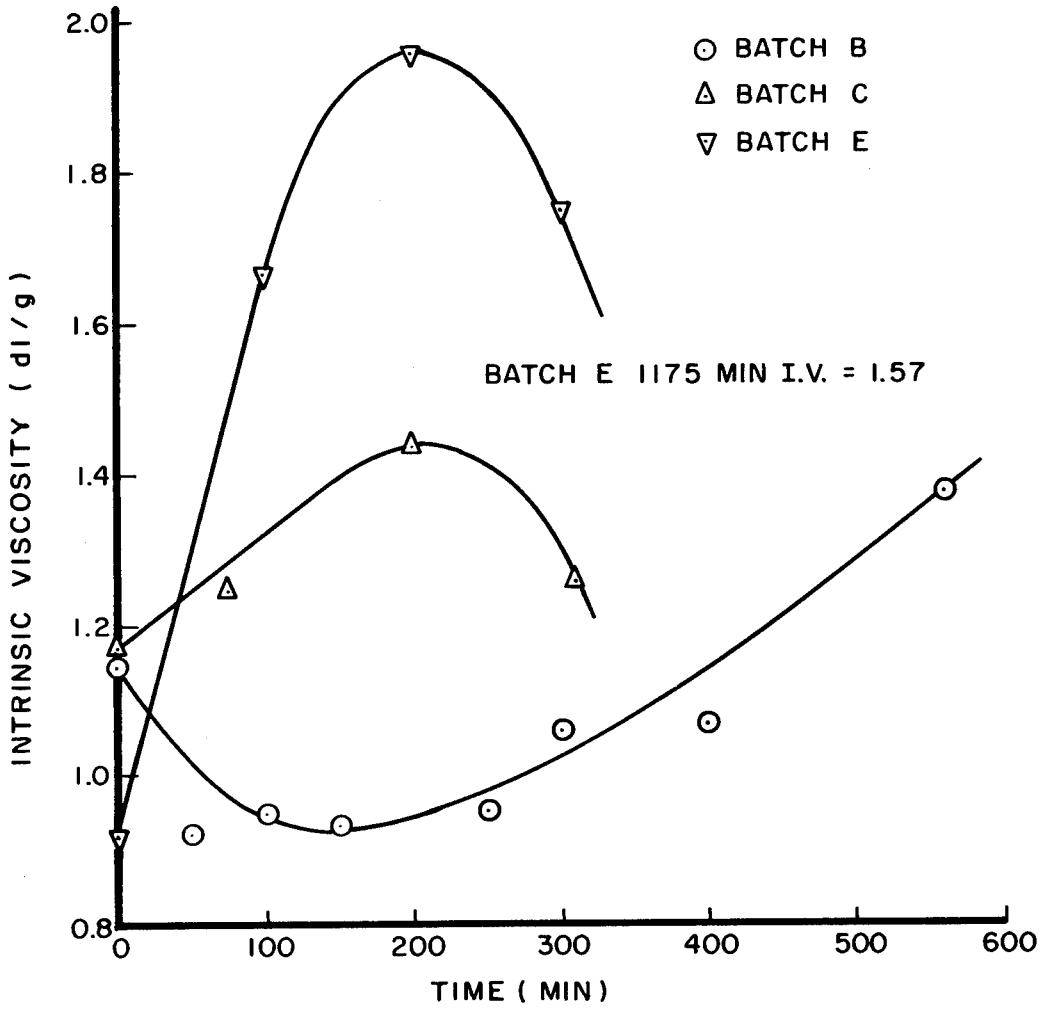


Figure 2. Intrinsic Viscosity Changes in Nylon 6.10 at 289 ± 1°C

Figure 3 shows the effects of exposure at a lower temperature, 280°C. In this case, the I.V. increases with time for all the batches. In 100 minutes, the I.V. of batch E increases to 1.41 as opposed to 1.67 at 289°C. It would seem that at the lower temperature and up to at least 300 minutes, further condensation is the predominant process, scission not occurring to any appreciable extent. The data shown in Figure 4 would seem to conflict with this however. Here, the variation of I.V. with time is shown for three temperatures for batch X, and in all cases the I.V.'s are significantly above their original values. A predrying cycle of 30 minutes at 220°C under high vacuum was carried out for all the experiments shown in Figure 4 in an attempt to remove the last traces of water from the polymer. No change in I.V. was noted for polymer subjected to this treatment alone.

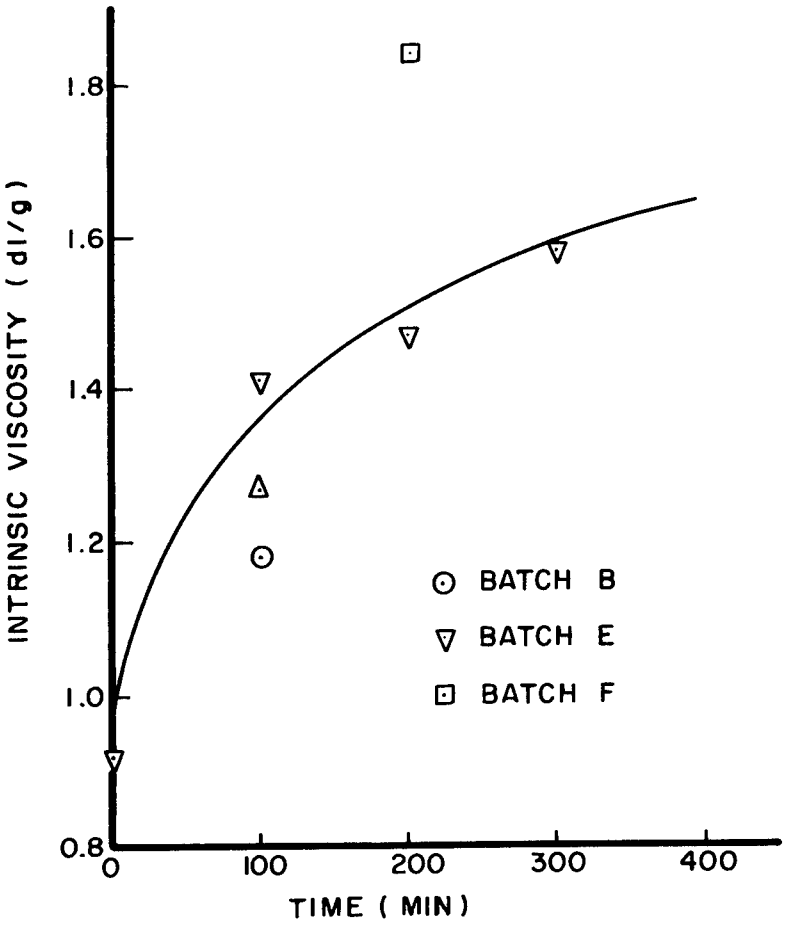


Figure 3. Viscosity Changes of Nylon 6.10 at 280°C

These data show, if nothing else, that it is essential to use a single standardized batch of polymer for all experiments in order to be able to compare results of viscosity studies. Further, the possible occurrence of further condensation, distillation of low molecular weight volatiles, branching and cross-linking will complicate interpretation of results.

3. END GROUP TITRATIONS ON NYLON 6.10

To determine unambiguously the molecular weight of a material a method which requires no calibration and which measures a colligative property is needed. End group titrations offer this possibility by counting, in the case of polyamides, the numbers of active-NH_2 and -COOH end groups. This method obviously requires that there be no end groups other than -COOH or -NH_2.

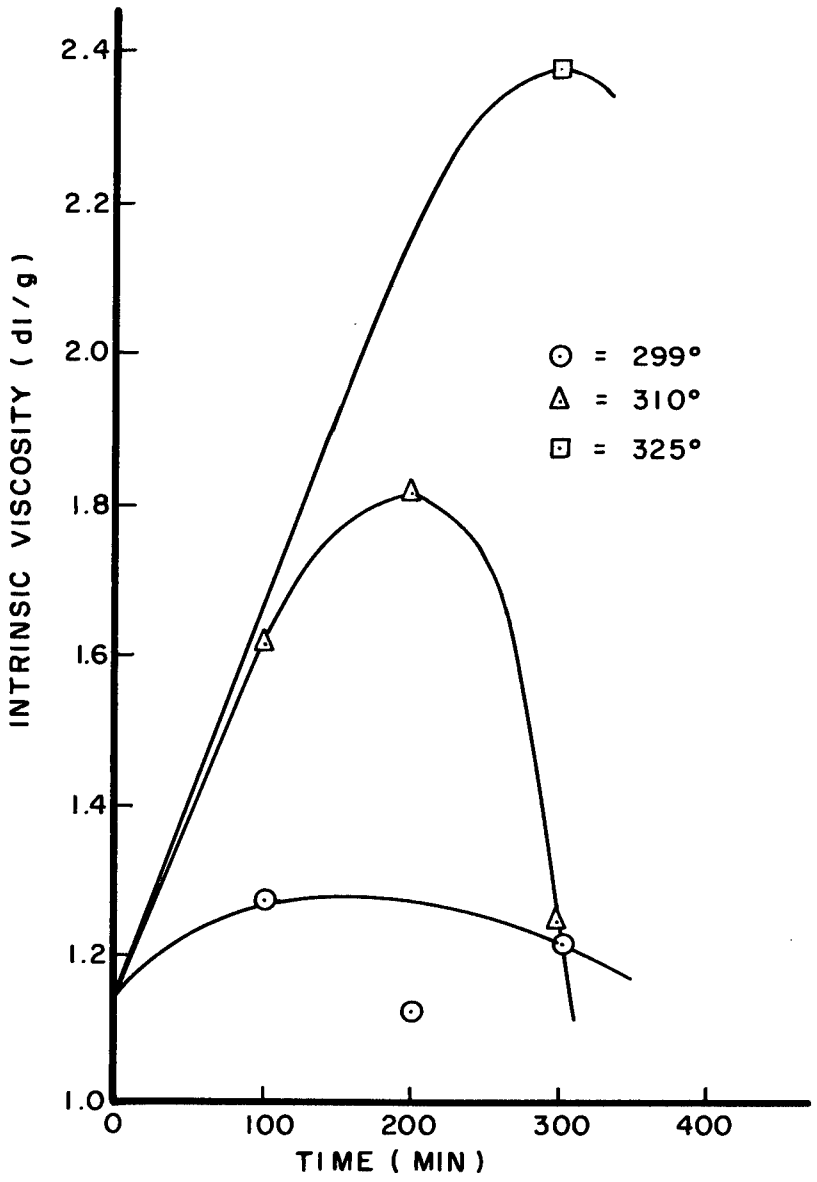


Figure 4. Viscosity Changes of Nylon 6.10

Since it was believed that the nylon 6.10 used here was prepared without the addition of end capping reagents (e.g., acetic acid) titration of m-cresol solutions of the polymer with dilute alcoholic HCl and NaOH solutions was attempted. The end point was determined by the break in the curve of conductivity against volume of titrant added. The results obtained were

$\overline{M}_n$ (NH_2) = 18.6 and 19.2 x 10^3 Average = 18.9 x 10^3

$\overline{M}_n$ (COOH) = 11.8 and 12.2 x 10^3 Average = 12.0 x 10^3

Differences in the numbers of each type of end group might be expected in low molecular weight polymers.

4. VAPOR PRESSURE OSMOMETRY

Several types of fluoroalcohols have been used for VPO measurements on polyamides (Reference 24). Both trifluoroethanol (TFE) and heptafluorobutanol were tried here and both were found to be suitable, the former at 37° and the latter at 65°C.

Meta-cresol was tried using the high temperature VPO chamber but the solvent attacked the insulation material around the chamber. It has been reported (Reference 32) that formic acid may be used if the chamber is gold plated.

Using TFE, solutions of nylon 6.10 up to a concentration of about 20 g/l may be prepared and used. Solutions above this concentration may also be prepared but separation tends to occur after several hours producing gel-like material which readily clears on being heated.

Figure 5 shows a plot of the reduced resistance against the concentration for solutions of nylon 6.10 in TFE at 37°C. An upswing in the curve is apparent at low concentrations but the linear part of the curve, when extrapolated to zero concentration yields a number average molecular weight of 7100.

The molecular weight of the nylon 6.6 used (Figure 6) is 4900.

The number average molecular weights of the degraded polymers referred to in Figure 4 were measured in heptafluorobutanol at 65°C. The results are summarized in Table I.

Inspection of this data allows no apparent correlation between the I.V. and $\overline{M}_n$ of the degraded materials; increases in I.V. are not always accompanied by increases in $\overline{M}_n$. If branching is taking place during degradation comparison of I.V.'s and $\overline{M}_n$ would not be valid since a single relationship between the two (the Mark Houwink equation) would not apply. A clue to this is given by the fact

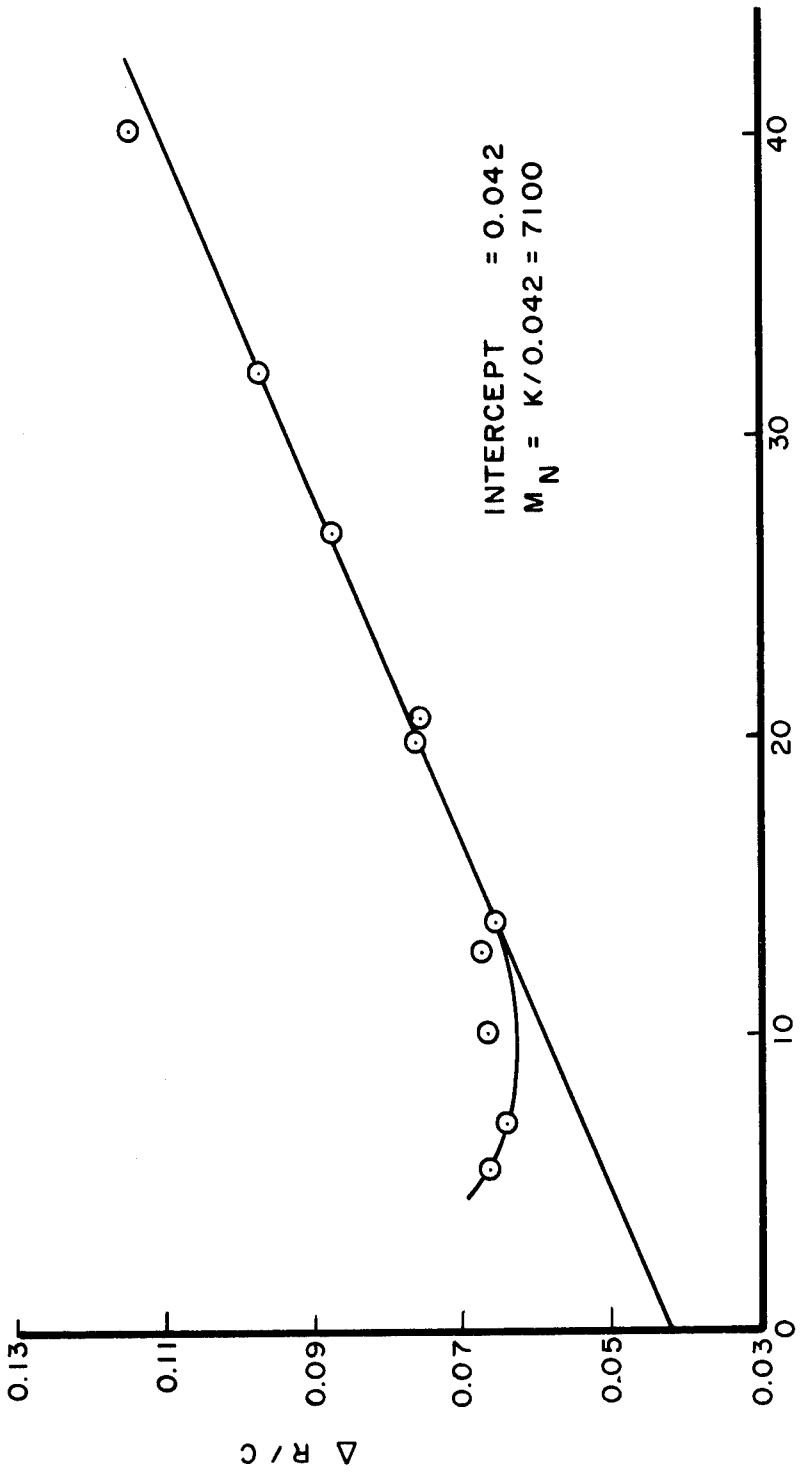


Figure 5. VPO Data for Nylon 6.10 in Trifluoroethanol at 37°C

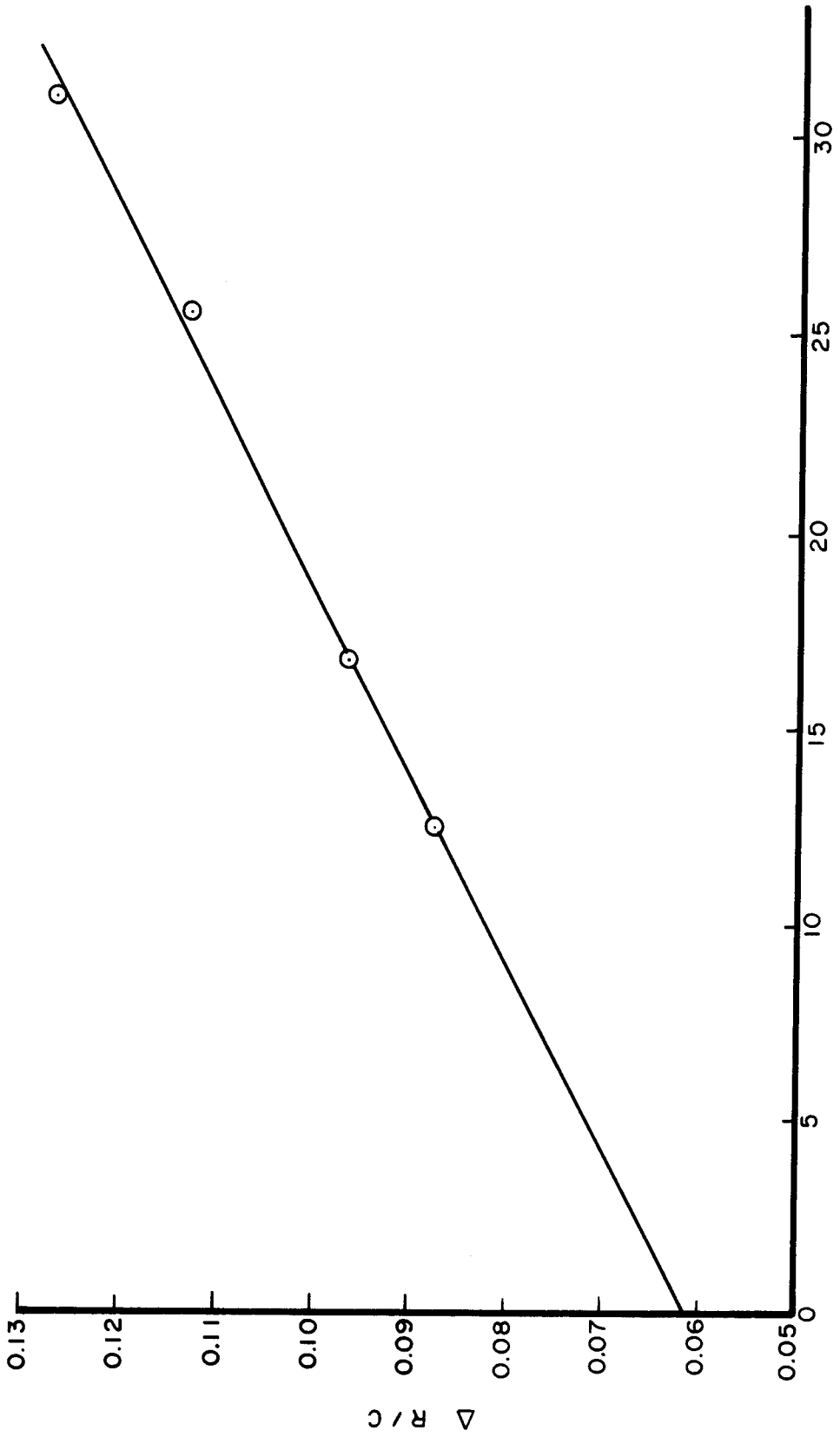


Figure 6. VPO Data for Nylon 6,6 in Trifluoroethanol at 37°C

TABLE I

INTRINSIC VISCOSITIES AND NUMBER AVERAGE MOLECULAR WEIGHTS OF NYLON 6.10

Exposure Time (min)	Exposure Temperature (°C)	Intrinsic Viscosity (dl/g)	M_n by VPO (Heptafluorobutanol at 65°C)
Original Material		1.15	7100*
100	299	1.28	8900
200	299	1.13	5600
300	299	1.22	10,700
100	310	1.62	5400
200	310	1.82	15,700
300	310	1.25	----
300	325	2.38	9300

* Determined in both heptafluorobutanol at 65° and trifluoroethanol at 37°C

that a plot of log I.V. versus log M_n using the data given in Table I is extremely scattered so it is not possible to derive a Mark-Houwink equation to fit the data.

5. MOLECULAR WEIGHT DISTRIBUTION BY GEL PERMEATION CHROMATOGRAPHY

Measurements of molecular weight distribution by Gel Permeation Chromatography (GPC) were made at Battelle Memorial Institute using 2,2,2-trifluoroethanol as solvent (Reference 33). Initially three columns designated 10^5, 10^4, and 10^4 were used for the GPC analysis. Figure 7 shows the distribution curves obtained for nylon 6.10 both undegraded and after exposure at various temperatures for differing times. The curves for the undegraded and for several of the degraded polymers exhibit single maxima but the sample exposed at 317°C for 500 minutes shows a pronounced double peak. The sample exposed at 327°C for 100 minutes shows slight evidence for a binodal distribution.

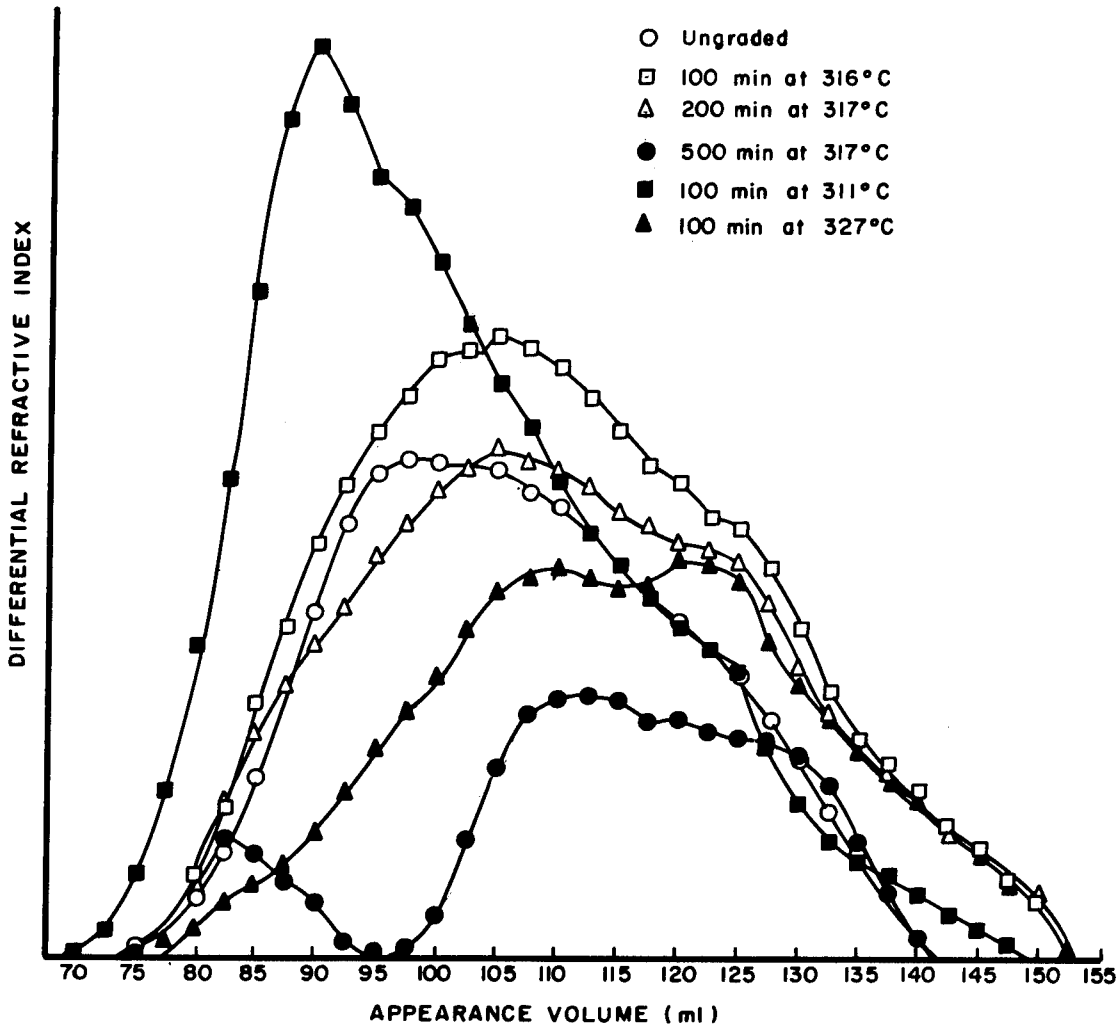


Figure 7. GPC Curves for Nylon 6.10

When nylon 6.10 was exposed to the mildest degradation (100 minutes at 311°C) there is a pronounced increase in the position and the height of the maximum. This may be attributed to either both sublimation of low molecular weight material,and further condensation of reactive end groups.

Similar data for nylon 6.6 is given in Figures 8 and 9. In Figure 8 the curve for the undegraded polymer is somewhat irregular and the various thermal exposures in all cases increase the molecular weight of the peak maximum.

The second series of distributions of nylon 6.6 (Figure 9) was conducted after one of the 10^4 GPC columns had been replaced by a 10^6 column. The undegraded material was then clearly demonstrated to be of binodal distribution. Exposure at 259°C for 100 minutes (polymer did not melt) caused a dramatic change in distribution. A single maximum was observed at $\overline{M}_n \cong 50,000$, the curve having a slight shoulder at a lower molecular weight. The distributions for samples held at 280° and 285°C for 100 minutes also had single maxima but at $\overline{M}_n \cong 19,000$.

Number average molecular weights of several of the nylon 6.6 polymers were measured by VPO in TFE solvent but there were inconsistencies between the values obtained and the positions of the maxima in the GPC traces.

The GPC data show that large changes in molecular weight distribution occur during thermal exposure of both nylon 6.6 and nylon 6.10 even under mild conditions. Normally an increase in the position of the maximum is evident but increased thermal exposure or higher temperatures cause gradual decreases in the molecular weight of the residues. The GPC data give a clue to the difficulties encountered in measuring number average molecular weights of degraded polymers especially using different batches of original polymer. Slight differences from batch to batch in the content of low molecular weight components will cause large differences in the molecular weight after thermal exposure.

Better removal of the low molecular weight material and end capping of residual reactive end groups from the original polymer is indicated for further studies of molecular weight changes.

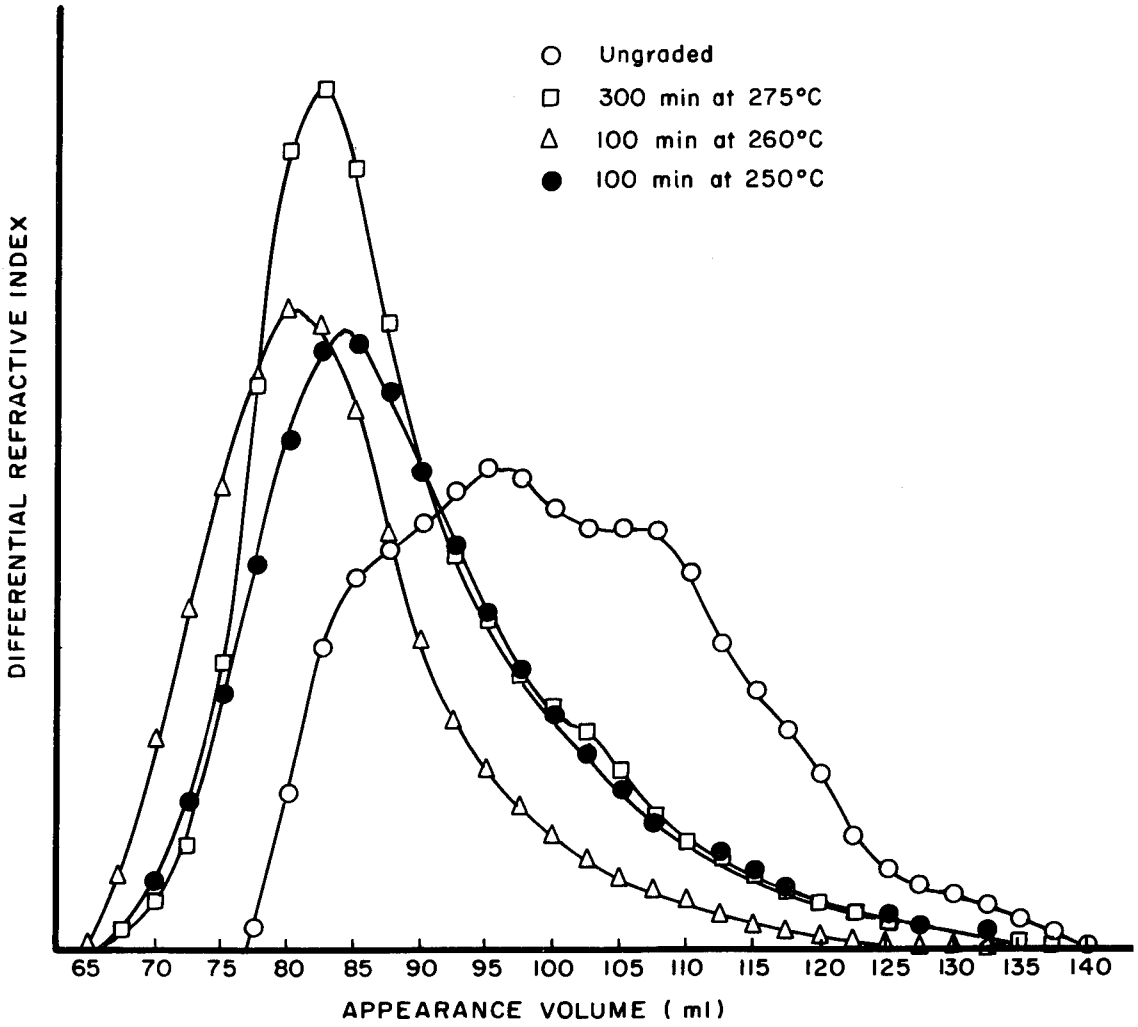


Figure 8. GPC Curves for Nylon 6.6

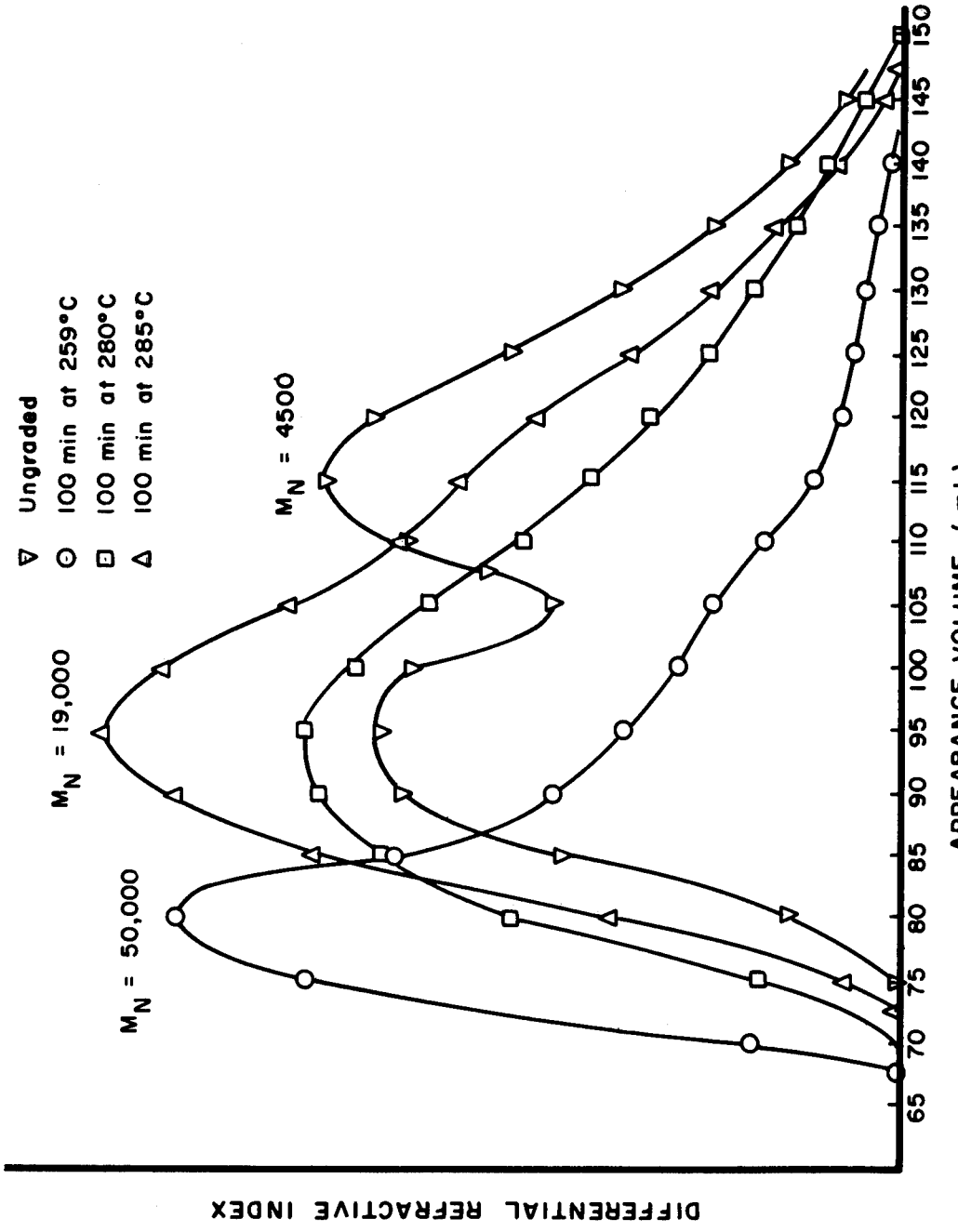


Figure 9. GPC Curves for Nylon 6.6

SECTION V

WEIGHT LOSS STUDIES

A previous report (Reference 10) describes determinations of the activation energy (E_a) for the thermal, vacuum weight loss of 500 mg samples of nylon 6.6 and nylon 6.10 under isothermal conditions. In our weight loss studies much importance has recently been placed on the determination of thermodynamic parameters from thermogravimetric experiments conducted under linearly increasing temperature profiles. It was deemed worthwhile, therefore, to compare the results obtained by the two methods. Since programmed temperature thermogravimetric experiments are more easily conducted (no temperature control problems such as those encountered in isothermal thermogravimetry), and since useful data may be obtained from the onset of degradation, it was hoped that the comparison would prove the worth of programmed thermogravimetry for the elucidation of mechanism and the determination of thermodynamic parameters involved in weight loss processes.

In other reports (References 8 and 9), we have described computer methods for the calculation of Arrhenius parameters from both isothermal and programmed temperature thermogravimetry data. The computational methods described were used for the results reported here.

To investigate the importance of diffusion controlled processes further isothermal experiments were carried out using various sample sizes.

1. NYLON 6.6

Some of the isothermal data presented in Reference 10 obtained using 500 mg samples has been recalculated using the computer method (Reference 9). The rate of weight loss data for this and other sample weights is collected in Appendix I. Figure 10 shows the variation of activation energy with percent weight loss using the recalculated 500 mg results. The original results quoted in Reference 10 are also shown for comparison. The differences may be attributed to the fact that only the higher temperature data was recalculated. Figure 10

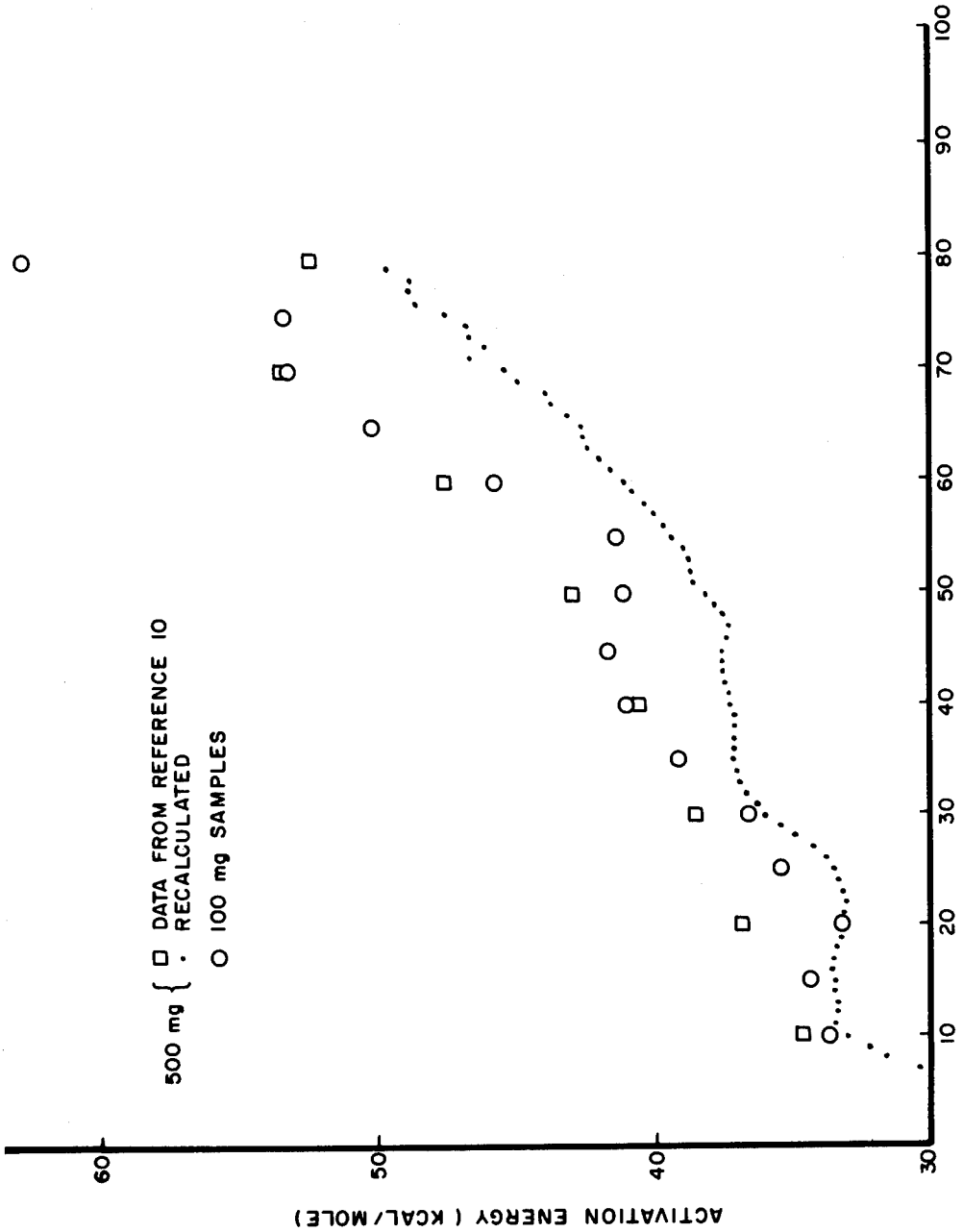


Figure 10. Variation of Activation Energy With Conversion for Different Sample Sizes of Nylon 6.6 (Isothermal Experiments)

also shows results obtained using 100 mg samples. The differences between the E_a values for 100 mg and 500 mg samples are small indicating that diffusion controlled weight losses either are unimportant or do not have significant effect on the activation energy. There is a small dependence of rate of weight loss (% per minute) on sample size (Figure 11). The rates for the small samples are slightly higher so some diffusion is occurring.

Programmed temperature rates of weight loss of 100 mg samples of nylon 6.6 were measured using heating rates ranging from 75°/hr to 450°C/hr and E_a was again determined as a function of the percent weight loss. Some of the rate data is given in Figure 12 and in Appendix II. The activation energy results obtained are plotted in Figure 13 where the isothermal 100 mg data is replotted for comparison. There is excellent agreement between the two sets of data for most of the degradation range. The maximum difference is about 8 kcal/mole at 30% weight loss. It would be difficult to specify the cause of the differences observed but it is probable that the programmed temperature data is more representative of the "true" activation energy for the weight loss process since there are no temperature stabilization difficulties.

In a pure single step chemical reaction the activation energy for a chemical change should remain constant throughout the reaction. Any change in E_a during the reaction is indicative of a change in the mechanism of that reaction. The significant changes in E_a with extent of weight loss observed here must therefore give an insight into the mechanism of the weight loss process. Up to 25% conversion E_a rises slowly. It then remains approximately constant until about 60% weight loss, and then increases continuously until the end of the reaction.

The early rise is probably due to early weight loss from evaporation of water either absorbed in the polymer or produced during condensation of the reactive end groups on the polymer chains. As condensation continues and the temperature rises, the importance of a higher activation energy process, namely, random chain scission, increases with consequent increase in E_a. At about 25% weight loss, condensation has been completed and the weight loss is due solely to loss of low molecular weight units produced during scission. The activation energy then remains constant until the rate of a third reaction becomes

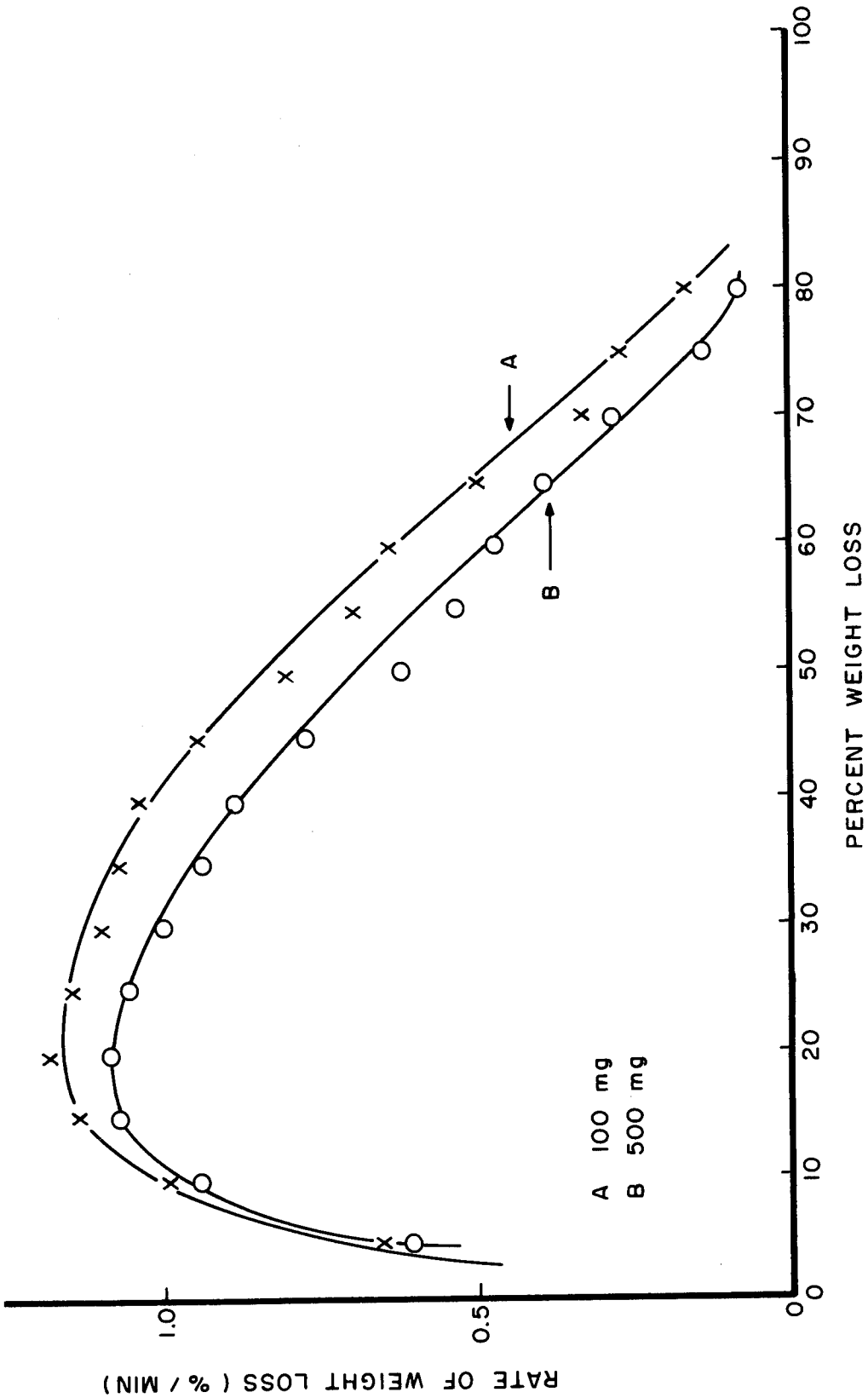


Figure 11. Variation of Rate of Weight Loss With Conversion for Two Sample Sizes of Nylon 6.6 at 380°C

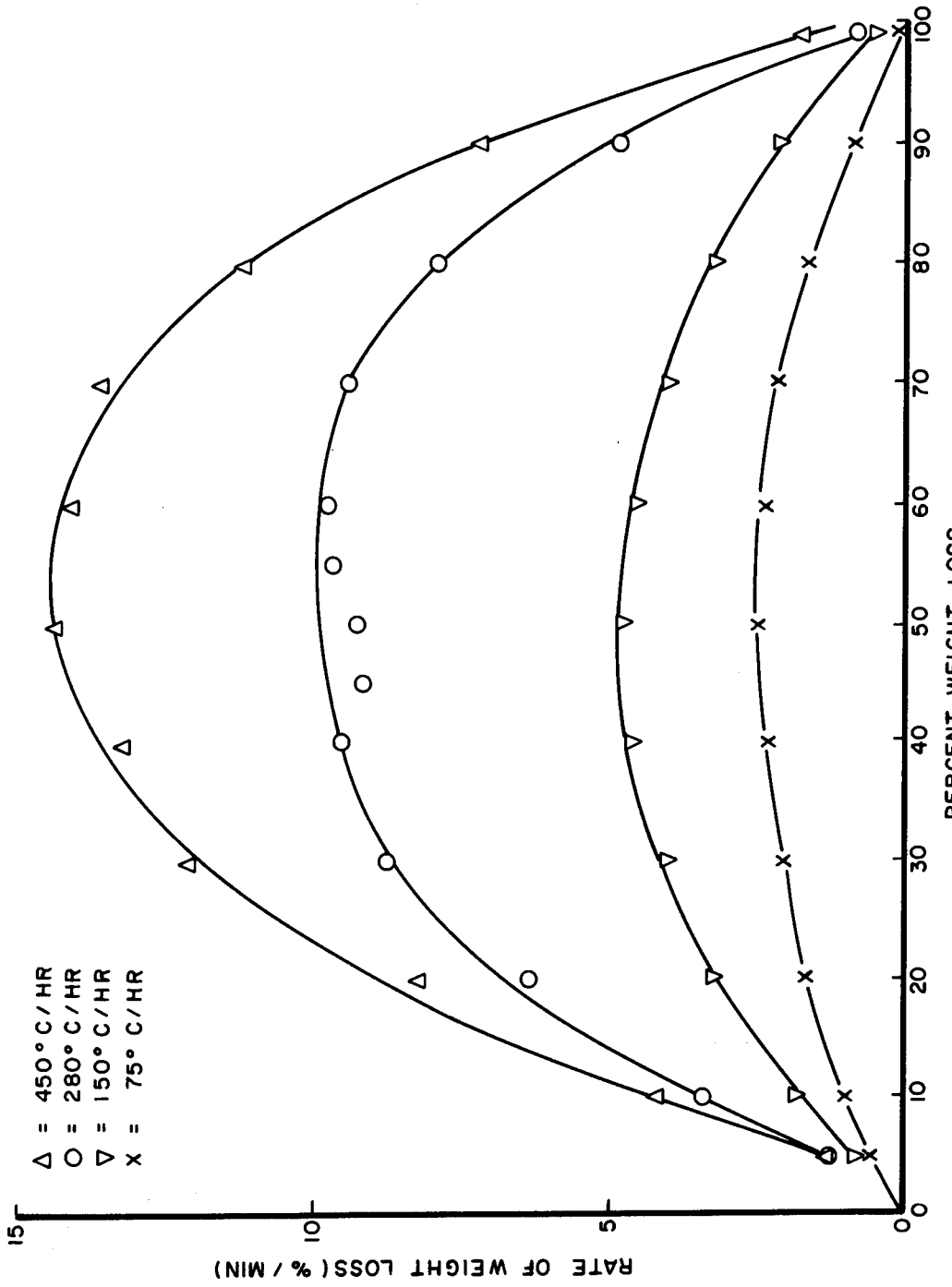


Figure 12. Variation of Rate of Weight Loss With Percent Weight Loss for Various Heating Rates for Nylon 6.6 (100 mg Samples)

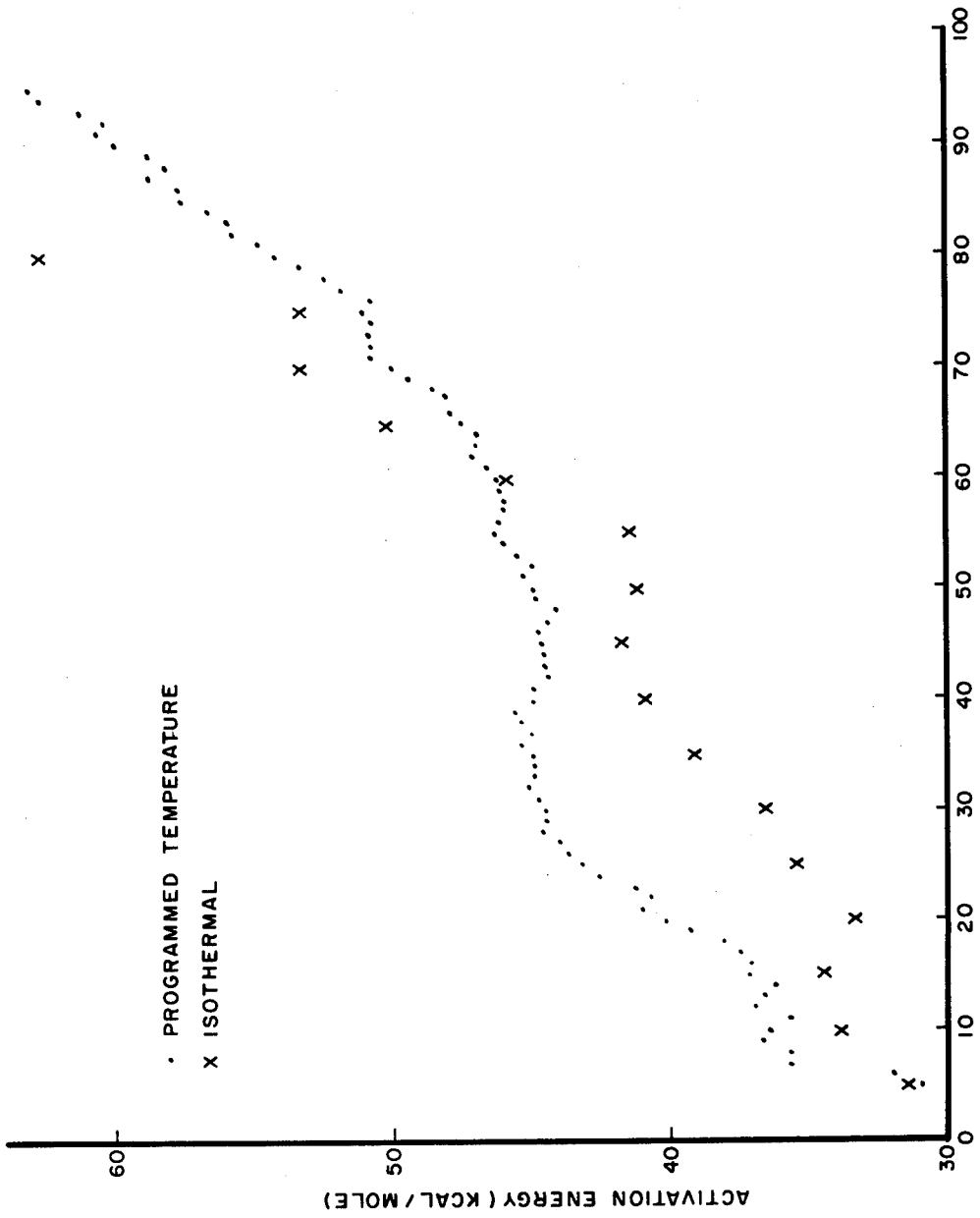


Figure 13. Activation Energy for Programmed Temperature Weight Loss of Nylon 6.6

appreciable. During degradation, nylon 6.6 readily cross-links forming a dark insoluble material. The activation energy required to remove a small volatile unit from a cross-linked material will be greater than for the corresponding straight chain material. Thus E_a would be expected to rise as the quantity of cross-linked polymer increases. This is shown up by the increase in E_a from 46 to 67 kcal/mole for the last 40% of the weight loss reaction.

Besides a change in E_a, there exists the possibility for a change in the apparent order of reaction for the weight loss. In Reference 8, it has been stressed that apparent order of reaction may not be the same as "order of reaction" in its classical definition but the term will still be used here.

The computer technique used here allows the determination of order of reaction from the slope of a plot of log A F(W) against log (percent weight remaining). Such a plot for the programmed temperature TG data for nylon 6.6 is shown in Figure 14. A good straight line having a slope of 1.16 may be drawn through the data representing weight loss from 25% to 80%. Thus, there is no significant change in the weight functionality of the rate of weight loss. The slight drop in the curve at low conversions tends to indicate that the process obeys random rather than "order" type kinetics. It has already been shown that the weight loss of this polymer obeys random kinetics since a true maximum can be observed in the rate of weight loss against weight loss curve during low temperature isothermal weight loss. The maximum isothermal rate of weight loss occurs in the range 20 to 30% weight loss compared with 25% (depending on the chain length of the evaporating molecule) predicted for random kinetics (Reference 34).

2. NYLON 6.10

Figure 15 shows the variation of activation energy with conversion for the isothermal weight loss of nylon 6.10. Line A represents the data originally given in Reference 10 using 500 mg samples. Lines B and C show data obtained with 250 mg and 100 mg samples, respectively. Here there are rather large differences in the E_a values at any given conversion and there does not seem to be a smooth trend in the value of E_a with change in sample size. Activation

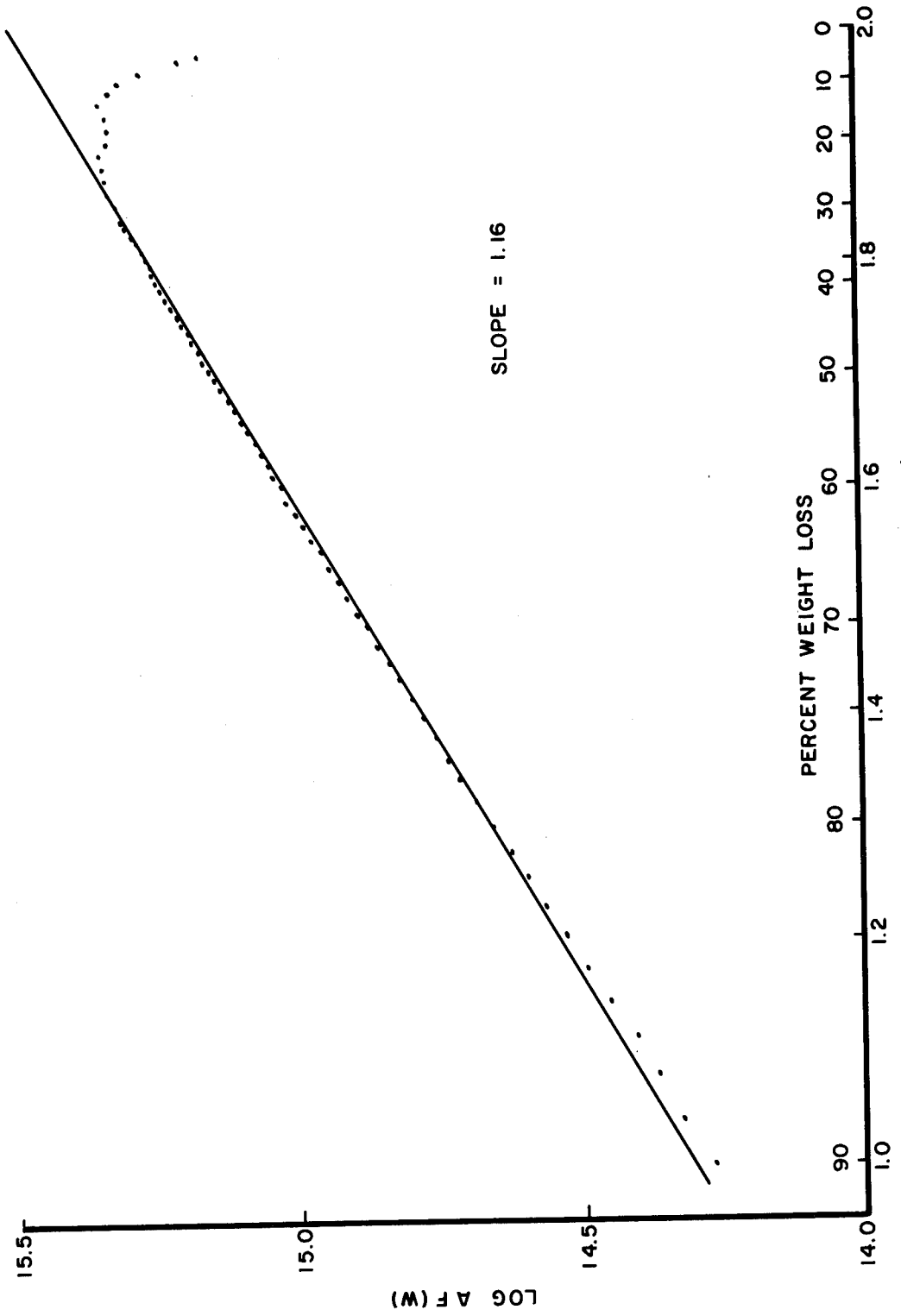


Figure 14. Log A F(W) Curve for Nylon 6.6 Weight Loss

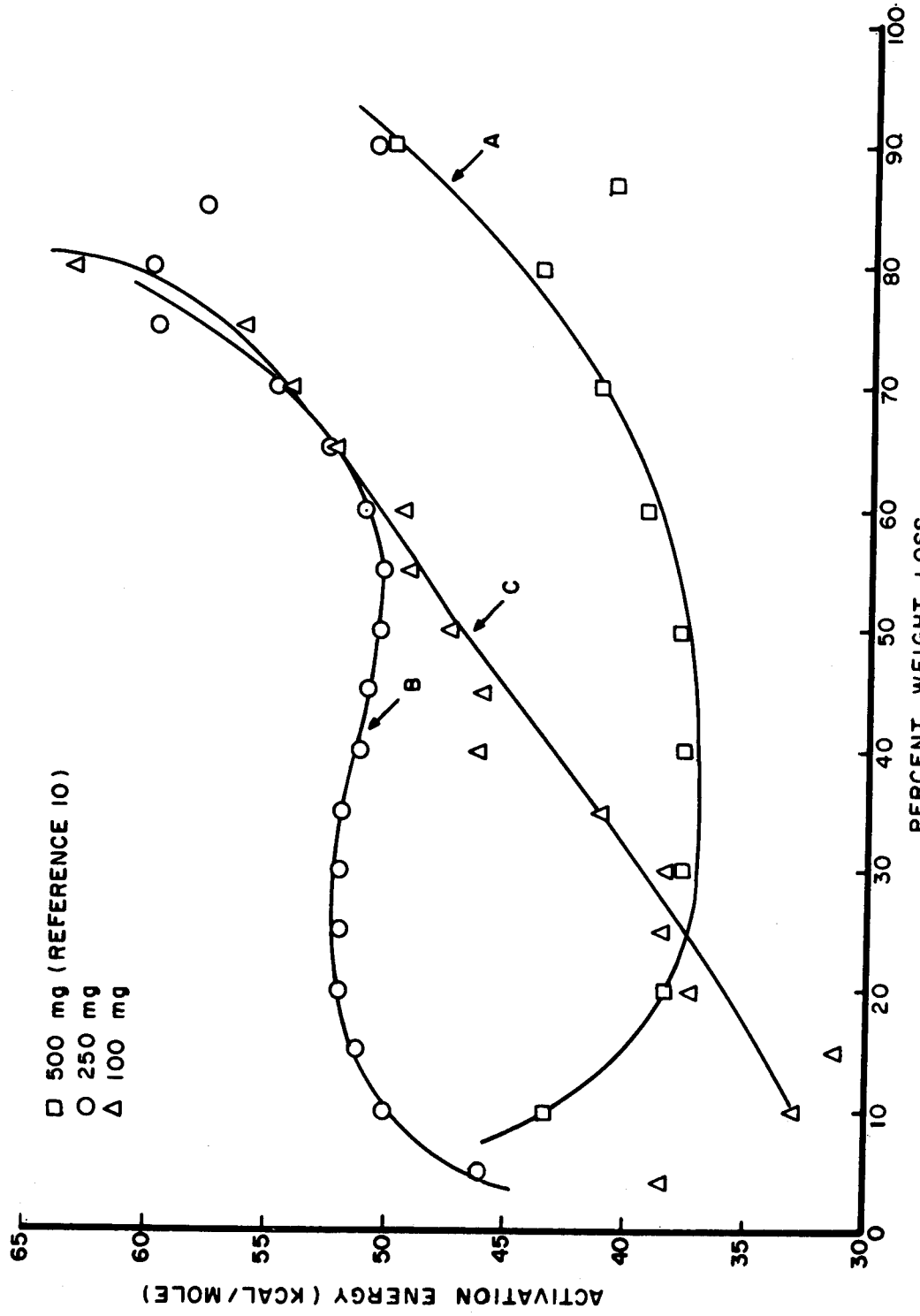


Figure 15. Variation of Activation Energy With Conversion for Different Sample Weights of Nylon 6,10 (Isothermal Data)

energies for the smaller samples agree reasonably well above 50% conversion. Any discrepancy in the low conversion region (up to about 20%) may be due to the failure to achieve temperature equilibrium until this amount of weight loss had occurred. The large differences between the 500 mg data and the smaller data show that diffusion controlled weight loss processes are taking place. For all further experiments with nylon 6.10, 100 mg was chosen as the standard sample size. For convenience the same weight of nylon 6.6 was also used.

Figure 16 shows the programmed temperature rate of weight loss data for 100 mg samples of nylon 6.10. The original data is given in Appendix II. The corresponding activation energies are given in Figure 17. Comparison with the isothermal data also shown in Figure 17 shows there are large differences between activation energies determined by the two different methods. The reasons for these differences are not known but they may be associated with the diffusion effects noted previously.

A further complication is that the programmed temperature data is gathered over a wider range of temperatures than is the isothermal data. This complication is inherent in the methods used and causes difficulties in interpretation of the results if the weight loss is not a simple process, e.g., if E_a varies with conversion or with temperature.

Below 20% weight loss the programmed temperature E_a increases rapidly and it then remains fairly constant at about 57 kcal/mole until total weight loss has occurred. The isothermal E_a, however, increases continuously during weight loss. Significantly, nylon 6.10 cross-links less readily than does nylon 6.6 so any increase in E_a due to cross-linking might be delayed until high weight losses have taken place.

Figure 18 shows the log A F(W) curve for nylon 6.10. The data representing 20 to 80% weight loss is represented by a good straight line having a slope of 0.98. A slight drop in the curve at low conversions is apparent showing the reaction is probably a random weight loss process.

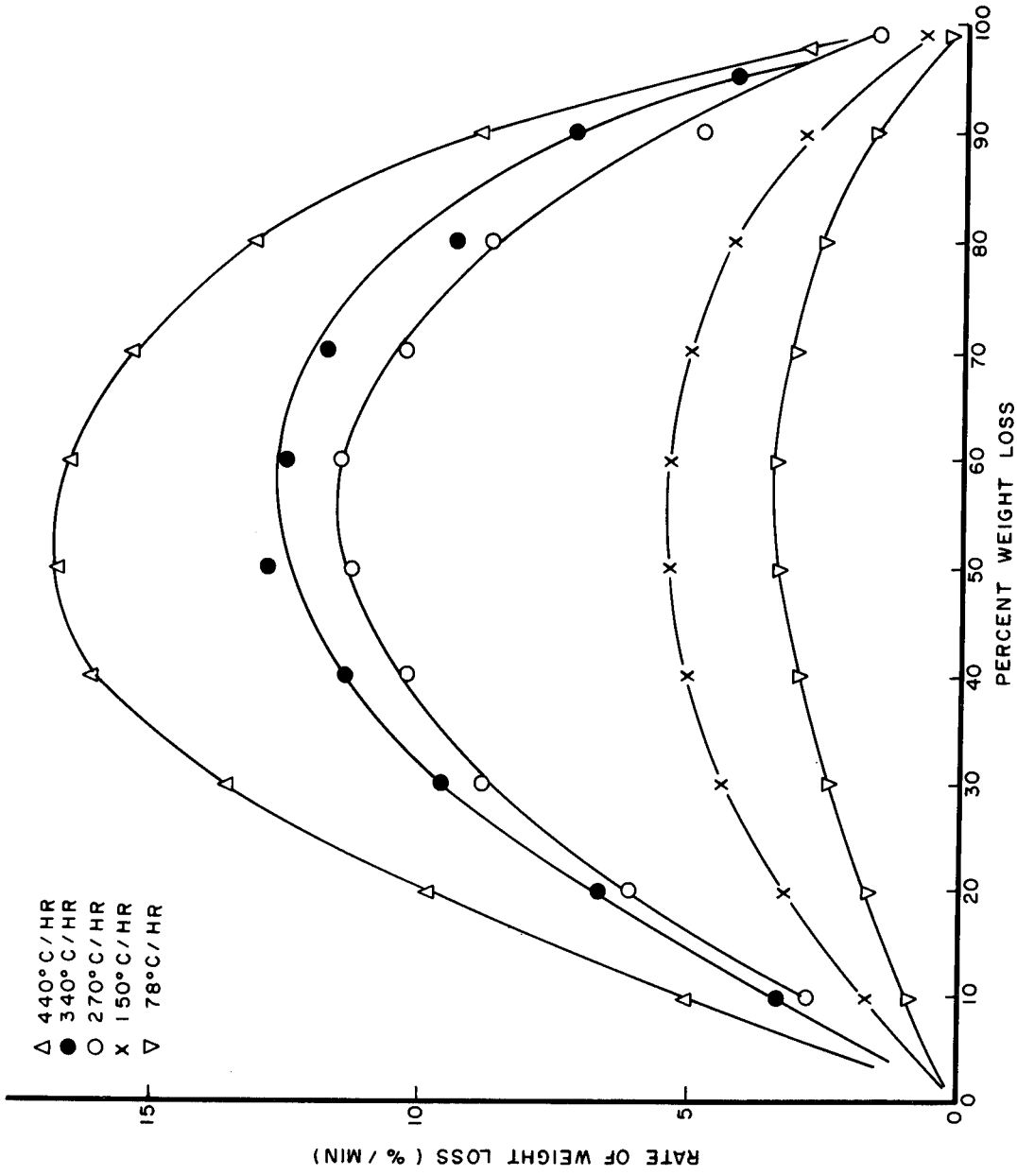


Figure 16. Variation of Rate of Weight Loss for Various Heating Rates for Nylon 6.10 (100 mg Samples)

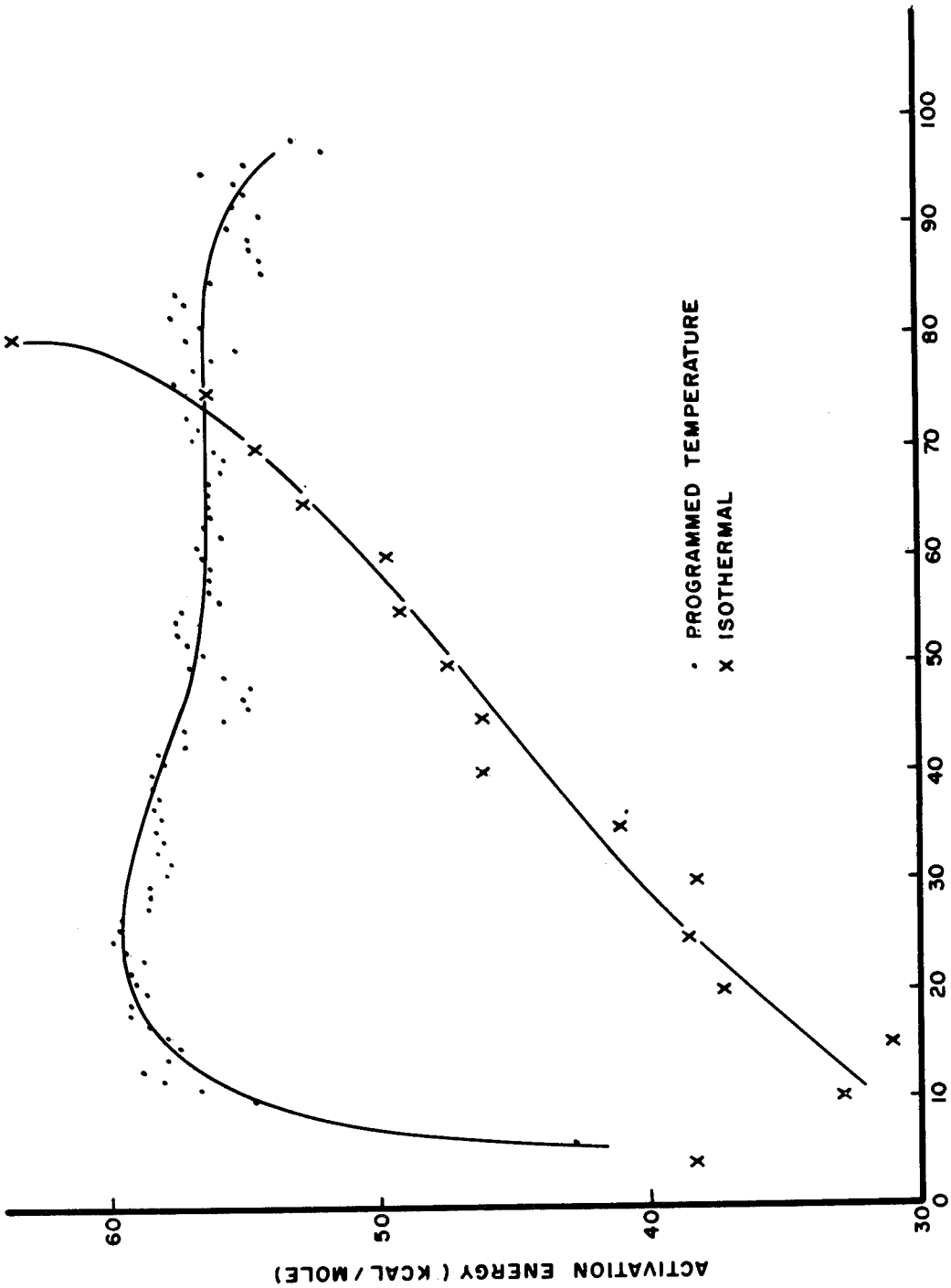


Figure 17. Activation Energy for Programmed Temperature Weight Loss of Nylon 6.10

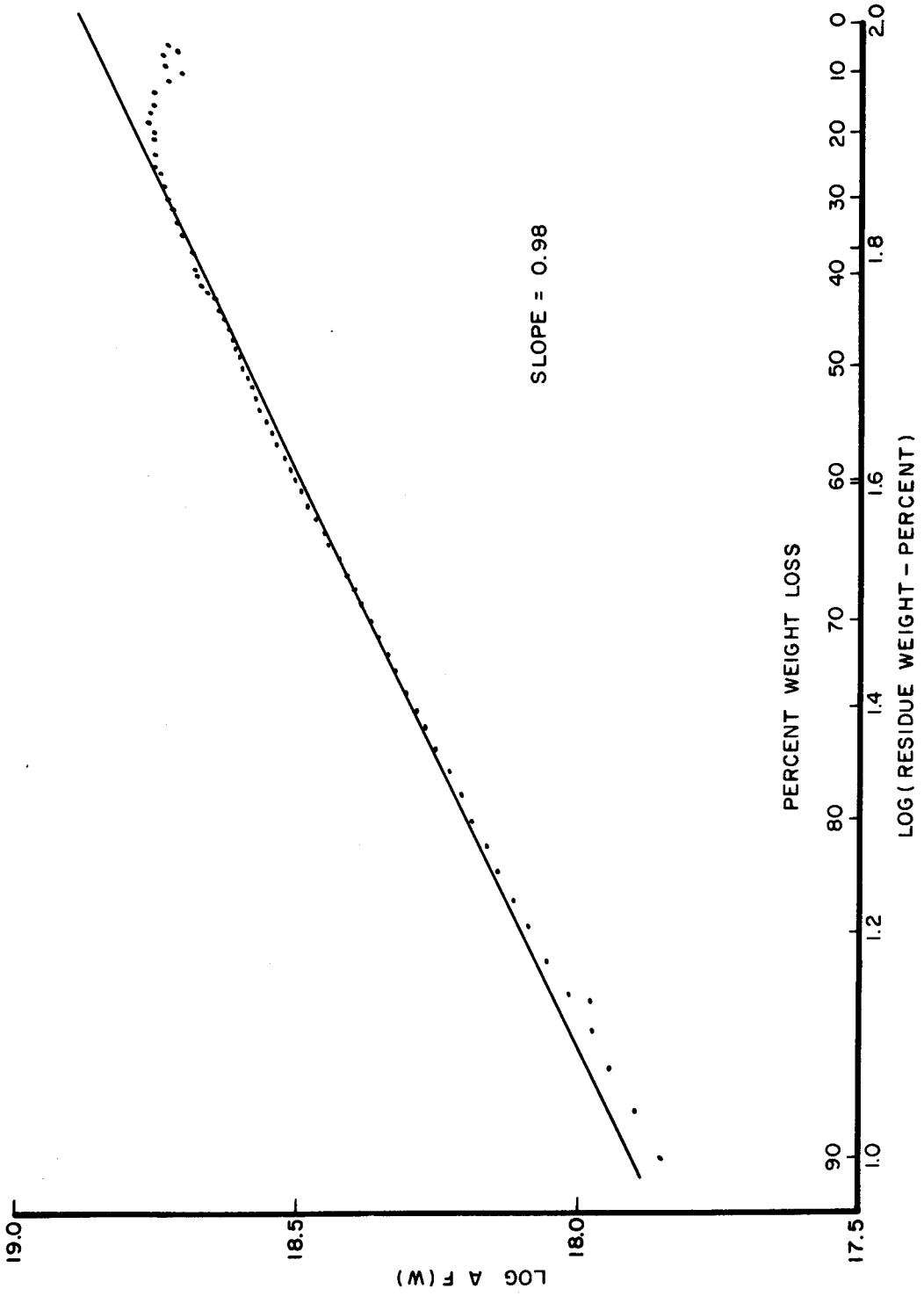


Figure 18. Log A F(W) Curve for Nylon 6.10 Weight Loss

SECTION VI

MASS SPECTROMETRIC THERMAL ANALYSIS

Small samples (usually about 1 mg) of both polymers were subjected to mass spectrometric thermal analysis (MTA) by the General Electric Company and a brief description of the results is given in Reference 35. A description of the experimental procedure is given in that reference. The method consists essentially of heating the sample from room temperature to 1000°C at a rate of 10°C per minute in a high vacuum. The effluent gases are pumped into the time-of-flight mass spectrometer and 200 preselected masses are scanned repetitively every 108 seconds. Computer data processing is used to obtain curves of ion intensity against temperature for each of the masses. It is hoped eventually, after suitable standard materials have been run, to be able to obtain complete quantitative analyses of the products of degradation.

1. NYLON 6.6

Examples of the original mass spectra are given in Reference 35. At about 425°C a peak is evident for many of the masses scanned and in most cases the peaks are well defined, dropping back rapidly to the base line. However, in some cases (e.g., mass 2, 13, 14, 24) a gradual increase in ion signal takes place above 600°C. At this temperature complete sample weight loss should have occurred so it is likely these are spurious signals caused by reevaporation of material which had condensed in the vacuum system, and can be ignored.

Figure 19 shows a bar graph of the peak intensities of all of the masses observed during MTA of Nylon 6.6. The compounds responsible for most of these peaks include NH_3, H_2O, CO, CO_2, cyclopentanone, and several hydrocarbons. It is difficult to obtain a quantitative analysis of all the compounds responsible for all of these peaks especially those present in small amounts.

Mass spectrometric data obtained from gases evolved from nylon 6.6 held isothermally were given in Reference 10. The same major components were present except for CO which was not trapped.

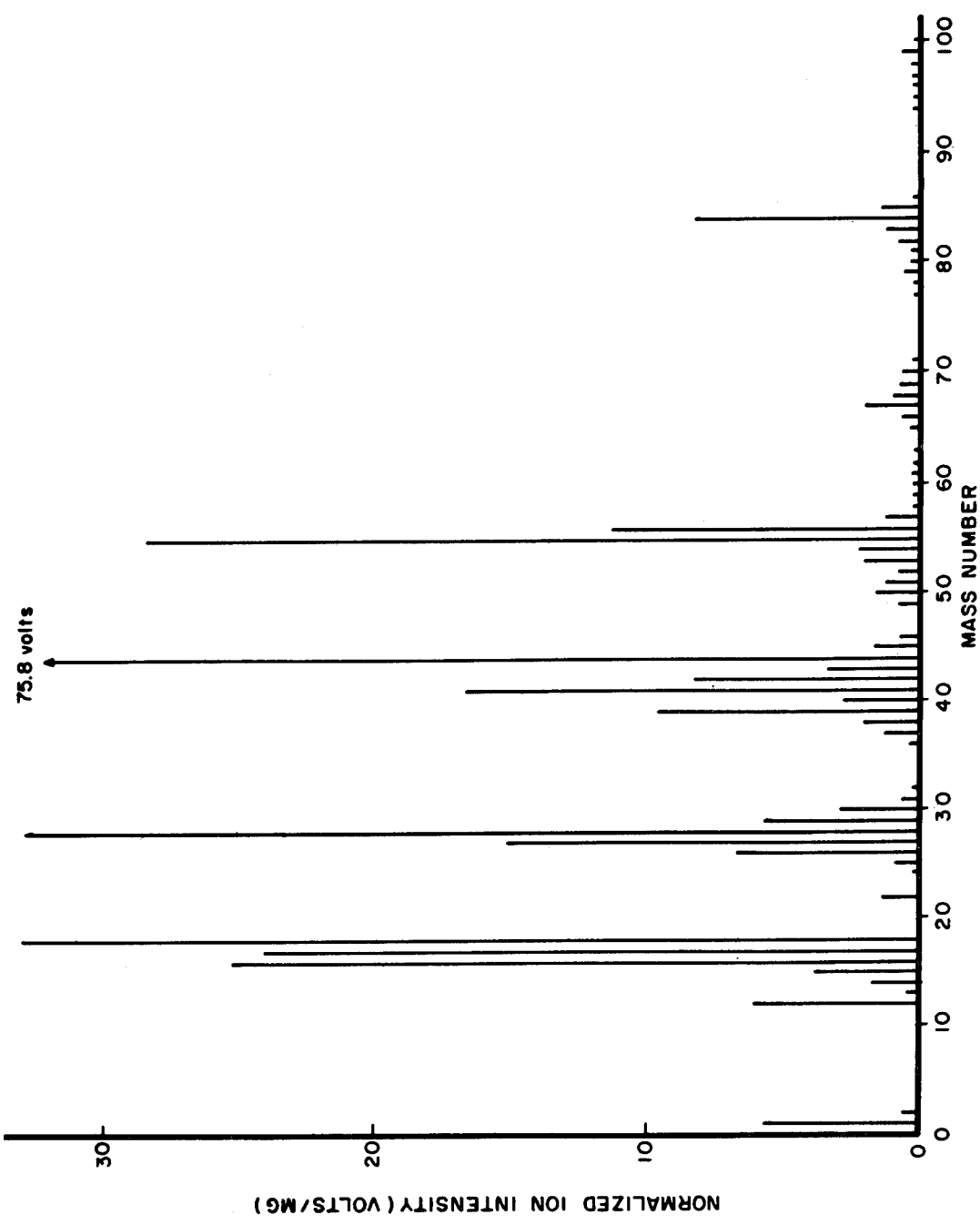


Figure 19. Maximum MTA Intensities for Nylon 6.6 Gaseous Degradation Products

2. NYLON 6.10

The bar graph for ion intensities of evolved gases is shown in Figure 20, and the major products were H_2O, CO, CO_2, 1,5-hexadiene and other hydrocarbons. The temperature for the maximum intensity is 450°C for this polymer and there is no evidence for significant gaseous evolution at lower temperatures. In contrast with the nylon 6.6 gaseous products, no ammonia was produced from nylon 6.10.

An obvious feature of these results is the presence of large quantities of water and CO_2 and CO in the gaseous products from both polymers. It is well known that water is strongly held by polyamides (probably by hydrogen bonding); some of the water detected probably was due to this effect, but further condensation would also give rise to water evolution. The source of CO_2 is probably decarboxylation of acid end groups.

Hydrocarbon fragments are derived from the aliphatic CH_2 chains and often occur as unsaturated compounds. The presence of cyclic ketones in the MTA data has not been confirmed but quantitative analysis of the data has yet to be attempted.

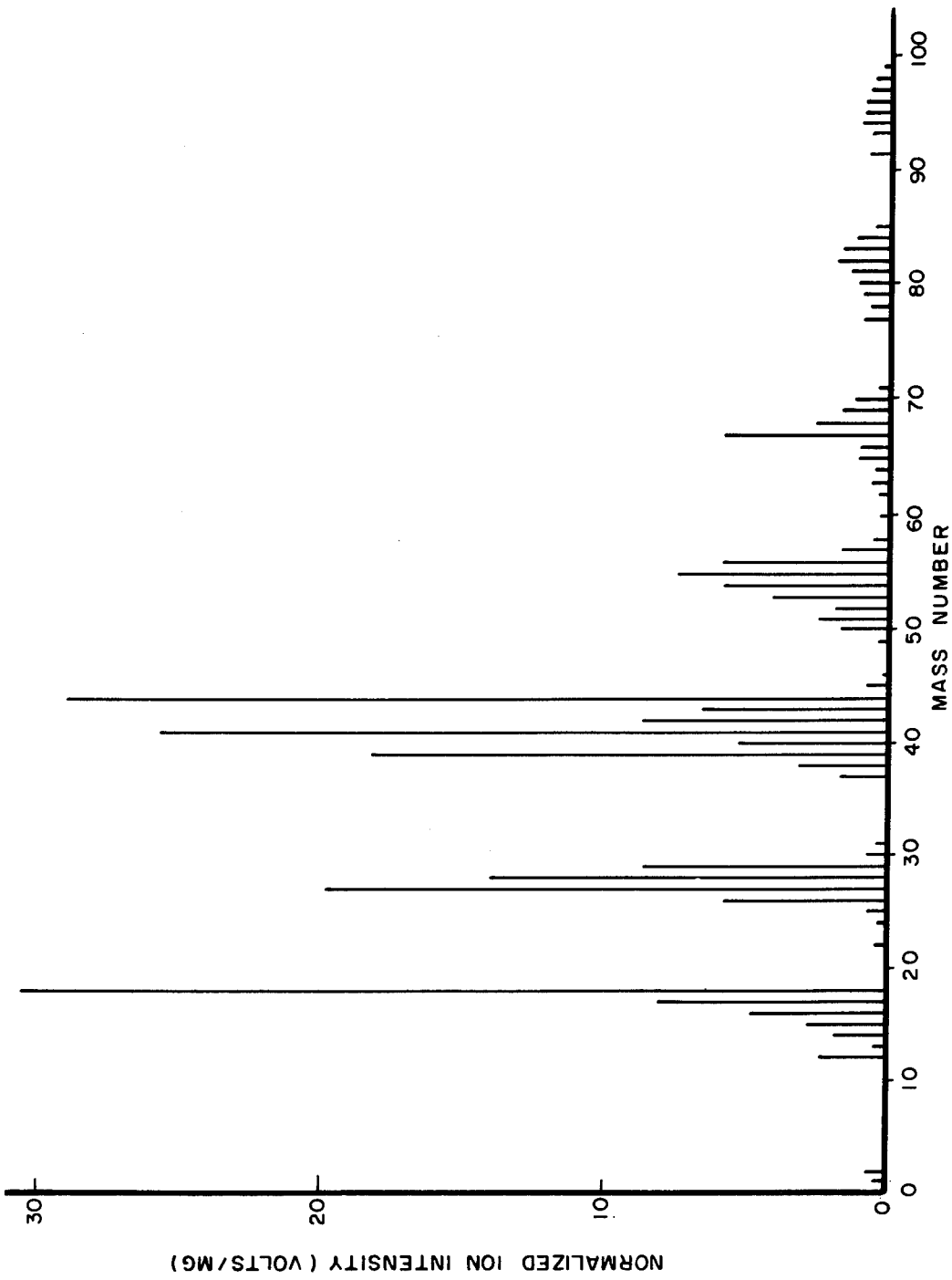


Figure 20. Maximum MTA Intensities for Nylon 6.10 Gaseous Degradation Products

SECTION VII

CONCLUSIONS

The molecular weight data presented in this report exemplify the difficulties in obtaining reproducible data from polyamides. The erratic behavior of polyamides has previously been attributed to the inability to obtain completely dry polymer samples (Reference 36). However, the present work indicates that the presence of low molecular weight material and polymerizable end groups in the polymer also contribute to the difficulties. The first of these has a profound effect on colligative solution properties of the polymers and the second complicates the interpretation of the molecular weight changes which take place during thermal exposure of these polymers, since polymerization takes place before scission or cross-linking. Attempts at removal of low molecular weight residues by extracting the polymer with solvents were evidently not successful. For further studies it would be desirable to employ narrow molecular weight range fractionated polyamides whose reactive end groups had been end capped.

These factors have, however, far less effect on the gathering of weight loss data. Obviously, polymers containing large amounts of low molecular weight fragments would not be desirable; small amounts would show up as slight early "bleeding" during weight loss experiments.

The removal of polymerizable end groups might also clarify some of the interpretation of mass spectral data, and could give further insight into the source or sources of water which is a major product. There are several possible sources for its production (desorption, further condensation, etc.) so removal of one of these would be useful.

The GPC data quoted demonstrates rather dramatically the large changes in molecular weight distribution of polymers which had been subjected even to mild thermal exposure.

The weight loss data obtained allows determinations of activation energy as a function of the amount of weight loss. This relationship combined with the A F(W) data permits mechanistic interpretation of the processes responsible for the overall weight loss.

Figure 13 which shows the activation energy data for nylon 6.6 weight loss indicates as explained previously, that E_a increases considerably during the weight loss but a plateau of 45 kcal/mole is evident in the range of weight loss from 30 to 60%. The early rise in E_a probably reflects further condensation of reactive end groups. The plateau region reflects the chain scission process which is apparent after the completion of further condensation. Finally the increase in E_a after 60% weight loss is caused by the various cross-linking reactions. These three processes may be summarized:

Further Condensation

$$R\cdots NH_2 + HOOC\cdots R' \longrightarrow R\cdots NHCO\cdots R' + H_2O \quad (1)$$

$$R\cdots NH_2 + H_2N\cdots R' \longrightarrow R\cdots NH\cdots R' + NH_3 \quad (2)$$

$$R\cdots COOH + HOOC\cdots R' \longrightarrow R\cdots CO\cdots R' + H_2O + CO_2 \quad (3)$$

Scission Reactions

$$R\cdots CONH(CH_2)_6\cdots R' \longrightarrow R\cdots CONH_2 + CH_2 = CH(CH_2)_4\cdots R' \quad (4)$$

$$R\cdots CONH_2 \longrightarrow R\cdots C\equiv N + H_2O \quad (5)$$

$$R\cdots CONH(CH_2)_6\cdots R' \longrightarrow R\cdots CON + CH_3(CH_2)_5\cdots R' \quad (6)$$

$$\downarrow$$

$$R\cdots N=C=O$$

$$\downarrow H_2O$$

$$R\cdots NH_2 + CO_2$$

Other reactions which break the hydrocarbon chains. (7)

Cross-Linking

$$R\cdots CO\cdots R' \text{ (from Equation 3)} + R\cdots NH_2 \longrightarrow R\cdots C(=N\cdots R)\cdots R' + H_2O \quad (8)$$

All these reactions have been suggested by Kamerbeek, et al. (Reference 15) who also detected several of the suggested intermediates by infrared spectrophotometry. The present mass spectrometric data confirms the evolution of NH_3, CO_2, and H_2O. Carbon monoxide production can be explained on the basis of homolysis of polymer chains on either side of a carbonyl group. The free radical produced will readily split out the stable CO molecule leaving hydrocarbon fragments. The mass spectra of both nylons are rather complex, the peaks occurring in clusters. The various hydrocarbon fragments are mainly responsible for these clusters but the homologous series of aliphatic nitriles is probably also present. These compounds might be produced in reactions of the type:

$$2R\ CH_2CONH_2 \longrightarrow RCH_2COOH + NH_3 + RCH_2CN \quad \text{(Reference 37)}.$$

The mechanism of this reaction involves the formation of a six-centered intermediate.

The changes in chemical reactions which are responsible for the changes in E_a as weight loss of nylon 6.6 proceeds are not reflected in the log A F(W) curve (Figure 14). The weight loss appears to be a random process even up to about 80% weight loss.

In the case of nylon 6.10, similar reactions may be postulated; however, the absence of NH_3 in the MTA data, if real, would obviously rule out reaction (2). The programmed temperature activation energy data for nylon 6.10 (Figure 17) shows E_a remains fairly constant above 20% weight loss. The slope of the log A F(W) curve is 0.98 and the downward curvature at low conversion tends to indicate a random weight loss is taking place.

In some cases, the weight loss data obtained under isothermal and under programmed temperature conditions did not agree. This is attributed to the interference of diffusion controlled processes, particularly in the case of nylon 6.10, showing the importance of using small sample sizes for study. Difficulties in isothermal temperature control and rapid heating of the sample to the degradation temperature may also have been involved.

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APPENDIX I

ISOTHERMAL NYLON 6.6 AND 6.10 RATE OF WEIGHT LOSS DATA

NYLON 6,6 ISOTHERMAL WEIGHT LOSS 500 MG.
RATE OF WT. LOSS IN PERCENT PER MINUTE

I.D.	IS17A	IS05A	IS02A	IS09A	IS06A	IS08A	IS03A	IS10A	IS04A	IS07A
TEMP.	319	347	353	355	360	367	373	375	380	385
PERCENT WT.LOSS	RATE OF WT.LOSS	RATE OF WT.LOSS	RATE OF WT.LOSS	RATE OF WT.LOSS	RATE OF WT.LOSS	RATE OF WT.LOSS	RATE OF WT.LOSS	RATE OF WT.LOSS	RATE OF WT.LOSS	RATE OF WT.LOSS
1	0.046	0.189	0.209	0.083	0.066	0.132	0.207	0.219	0.218	0.074
2	0.030	0.217	0.248	0.123	0.084	0.185	0.301	0.319	0.330	0.136
3	0.035	0.362	0.369	0.189	0.137	0.349	0.387	0.437	0.426	0.267
4	0.040	0.479	0.461	0.246	0.185	0.480	0.484	0.550	0.521	0.412
5	0.044	0.573	0.511	0.283	0.223	0.575	0.578	0.638	0.610	0.512
6	0.048	0.632	0.542	0.306	0.251	0.633	0.658	0.697	0.701	0.583
7	0.050	0.675	0.566	0.316	0.273	0.698	0.722	0.731	0.775	0.654
8	0.052	0.711	0.585	0.315	0.290	0.742	0.774	0.753	0.836	0.726
9	0.053	0.744	0.597	0.318	0.307	0.779	0.811	0.752	0.893	0.777
10	0.054	0.759	0.608	0.319	0.321	0.810	0.838	0.746	0.943	0.824
11	0.054	0.777	0.616	0.327	0.334	0.820	0.849	0.745	0.983	0.871
12	0.054	0.795	0.618	0.340	0.345	0.835	0.848	0.744	1.015	0.907
13	0.054	0.813	0.622	0.353	0.354	0.846	0.845	0.746	1.041	0.957
14	0.054	0.830	0.622	0.360	0.362	0.852	0.827	0.748	1.062	0.988
15	0.054	0.848	0.619	0.366	0.368	0.857	0.811	0.757	1.074	1.026
16	0.053	0.865	0.617	0.375	0.370	0.841	0.790	0.761	1.083	1.051
17	0.052	0.880	0.620	0.383	0.374	0.824	0.781	0.764	1.088	1.078
18	0.052	0.884	0.624	0.388	0.376	0.808	0.774	0.767	1.088	1.102
19	0.051	0.886	0.626	0.390	0.378	0.795	0.773	0.771	1.083	1.117
20	0.050	0.885	0.619	0.390	0.378	0.786	0.772	0.776	1.084	1.120
21	0.049	0.882	0.604	0.389	0.376	0.776	0.772	0.784	1.082	1.128
22	0.048	0.878	0.582	0.391	0.374	0.764	0.769	0.790	1.076	1.135
23	0.047	0.868	0.568	0.390	0.371	0.750	0.766	0.794	1.070	1.142
24	0.047	0.857	0.560	0.386	0.370	0.732	0.759	0.793	1.064	1.147
25	0.046	0.840	0.555	0.384	0.370	0.705	0.753	0.786	1.056	1.152
26	0.046	0.822	0.550	0.376	0.371	0.671	0.744	0.775	1.047	1.154
27	0.045	0.807	0.535	0.364	0.370	0.632	0.736	0.763	1.037	1.153
28	0.044	0.793	0.529	0.353	0.367	0.589	0.728	0.748	1.025	1.155
29	0.043	0.783	0.525	0.345	0.364	0.541	0.720	0.729	1.013	1.149
30	0.042	0.777	0.519	0.339	0.358	0.513	0.709	0.708	1.000	1.144
31	0.041	0.762	0.511	0.334	0.351	0.496	0.696	0.679	0.987	1.139
32	0.040	0.746	0.503	0.325	0.343	0.488	0.682	0.653	0.973	1.130
33	0.039	0.729	0.492	0.318	0.341	0.487	0.666	0.627	0.959	1.127
34	0.038	0.711	0.483	0.308	0.338	0.489	0.651	0.617	0.948	1.121
35	0.037	0.695	0.472	0.302	0.333	0.487	0.638	0.611	0.937	1.105
36	0.035	0.682	0.461	0.298	0.328	0.484	0.628	0.612	0.929	1.098
37	0.034	0.669	0.449	0.294	0.321	0.479	0.619	0.611	0.916	1.087
38	0.032	0.656	0.435	0.290	0.312	0.472	0.608	0.620	0.907	1.066
39	0.031	0.640	0.420	0.285	0.302	0.465	0.600	0.630	0.895	1.053
40	0.030	0.626	0.404	0.278	0.294	0.456	0.590	0.647	0.884	1.031
41	0.029	0.608	0.390	0.269	0.288	0.443	0.579	0.659	0.869	1.010
42	0.028	0.595	0.378	0.258	0.280	0.433	0.567	0.668	0.850	0.984

I.D.	IS17A	IS05A	IS02A	IS09A	IS06A	IS08A	IS03A	IS10A	IS04A	IS07A
TEMP.	319	347	353	355	360	367	373	375	380	385
PERCENT WT.LOSS	RATE OF WT.LOSS	RATE OF WT.LOSS	RATE OF WT.LOSS	RATE OF WT.LOSS	RATE OF WT.LOSS	RATE OF WT.LOSS	RATE OF WT.LOSS	RATE OF WT.LOSS	RATE OF WT.LOSS	RATE OF WT.LOSS
43	0.027	0.579	0.368	0.248	0.274	0.423	0.553	0.672	0.826	0.964
44	0.026	0.560	0.359	0.240	0.267	0.412	0.541	0.673	0.804	0.931
45	0.025	0.539	0.349	0.234	0.260	0.405	0.530	0.671	0.771	0.918
46	0.024	0.520	0.335	0.229	0.255	0.395	0.520	0.667	0.738	0.903
47	0.023	0.498	0.327	0.220	0.250	0.385	0.510	0.672	0.704	0.887
48	0.022	0.479	0.324	0.209	0.242	0.373	0.499	0.661	0.670	0.872
49	0.021	0.451	0.310	0.199	0.234	0.361	0.486	0.648	0.641	0.860
50	0.019	0.430	0.294	0.188	0.226	0.348	0.471	0.618	0.619	0.830
51	0.017	0.408	0.283	0.180	0.219	0.336	0.449	0.582	0.601	0.813
52	0.015	0.392	0.273	0.176	0.213	0.324	0.431	0.536	0.583	0.801
53	0.013	0.372	0.260	0.173	0.208	0.313	0.415	0.516	0.565	0.788
54	0.012	0.359	0.253	0.168	0.201	0.304	0.403	0.494	0.548	0.774
55	0.013	0.347	0.240	0.162	0.193	0.295	0.393	0.480	0.533	0.759
56	0.012	0.334	0.223	0.155	0.186	0.287	0.386	0.467	0.521	0.742
57	0.011	0.315	0.214	0.147	0.178	0.278	0.370	0.454	0.509	0.725
58	0.011	0.301	0.204	0.140	0.171	0.267	0.357	0.437	0.498	0.708
59	0.	0.287	0.	0.134	0.162	0.252	0.343	0.424	0.487	0.690
60	0.	0.268	0.	0.128	0.155	0.235	0.331	0.405	0.473	0.669
61	0.	0.253	0.	0.122	0.147	0.219	0.320	0.383	0.457	0.647
62	0.	0.237	0.	0.116	0.138	0.205	0.309	0.362	0.441	0.628
63	0.	0.221	0.	0.110	0.130	0.194	0.298	0.344	0.425	0.605
64	0.	0.204	0.	0.	0.123	0.187	0.283	0.319	0.408	0.581
65	0.	0.186	0.	0.	0.117	0.182	0.267	0.299	0.395	0.555
66	0.	0.170	0.	0.	0.108	0.173	0.251	0.289	0.377	0.530
67	0.	0.157	0.	0.	0.101	0.161	0.238	0.269	0.357	0.501
68	0.	0.	0.	0.	0.096	0.150	0.226	0.247	0.339	0.475
69	0.	0.	0.	0.	0.085	0.139	0.214	0.235	0.316	0.453
70	0.	0.	0.	0.	0.079	0.128	0.211	0.213	0.283	0.428
71	0.	0.	0.	0.	0.071	0.118	0.194	0.189	0.230	0.405
72	0.	0.	0.	0.	0.	0.108	0.162	0.176	0.171	0.384
73	0.	0.	0.	0.	0.	0.102	0.150	0.166	0.156	0.363
74	0.	0.	0.	0.	0.	0.096	0.142	0.152	0.147	0.343
75	0.	0.	0.	0.	0.	0.087	0.134	0.138	0.139	0.321
76	0.	0.	0.	0.	0.	0.078	0.120	0.	0.128	0.300
77	0.	0.	0.	0.	0.	0.	0.111	0.	0.115	0.277
78	0.	0.	0.	0.	0.	0.	0.103	0.	0.107	0.255
79	0.	0.	0.	0.	0.	0.	0.092	0.	0.101	0.231
80	0.	0.	0.	0.	0.	0.	0.	0.	0.087	0.205
81	0.	0.	0.	0.	0.	0.	0.	0.	0.074	0.184
82	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.166
83	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.149
84	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.130
85	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.107
86	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.089
87	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.089
88	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.080
89	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.070
90	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.060
91	0.	0.	0.	0.	0.	0.	0.	0.	0.	0.

NYLON 6,6 ISOTHERMAL WEIGHT LOSS 500 MG.
RATE OF WT. LOSS IN PERCENT PER MINUTE

ISO. TEMP.	IS11A 394	IS01A 400
PERCENT WT.LOSS	RATE OF WT.LOSS	RATE OF WT.LOSS
1	0.323	0.346
2	0.526	0.573
3	0.741	0.825
4	0.962	1.071
5	1.163	1.279
6	1.321	1.453
7	1.461	1.602
8	1.586	1.730
9	1.695	1.833
10	1.796	1.915
11	1.879	1.974
12	1.949	2.014
13	2.011	2.038
14	2.067	2.051
15	2.112	2.056
16	2.145	2.047
17	2.162	2.029
18	2.170	2.011
19	2.171	1.990
20	2.172	1.961
21	2.172	1.931
22	2.171	1.911
23	2.170	1.900
24	2.160	1.888
25	2.138	1.880
26	2.116	1.871
27	2.095	1.870
28	2.066	1.875
29	2.028	1.881
30	1.996	1.884
31	1.970	1.882
32	1.942	1.881
33	1.909	1.878
34	1.876	1.866
35	1.845	1.839
36	1.816	1.819
37	1.782	1.793
38	1.741	1.766
39	1.708	1.727
40	1.670	1.690
41	1.630	1.648

IJC.	IS11A	IS01A
TEMP.	394	400
PERCENT	RATE OF	RATE OF
WT.LCSS	WT.LCSS	WT.LOSS
42	1.590	1.607
43	1.550	1.571
44	1.505	1.532
45	1.459	1.497
46	1.410	1.452
47	1.358	1.426
48	1.314	1.391
49	1.266	1.354
50	1.229	1.312
51	1.199	1.284
52	1.159	1.257
53	1.135	1.232
54	1.105	1.208
55	1.080	1.185
56	1.052	1.157
57	1.019	1.125
58	0.982	1.089
59	0.951	1.052
60	0.917	1.012
61	0.890	0.970
62	0.859	0.927
63	0.831	0.890
64	0.795	0.850
65	0.762	0.811
66	0.728	0.774
67	0.693	0.741
68	0.653	0.703
69	0.619	0.667
70	0.589	0.631
71	0.557	0.602
72	0.527	0.574
73	0.504	0.544
74	0.477	0.510
75	0.449	0.479
76	0.421	0.447
77	0.403	0.416
78	0.377	0.382
79	0.348	0.354
80	0.312	0.327
81	0.284	0.286
82	0.254	0.252
83	0.228	0.233
84	0.199	0.209
85	0.174	0.187
86	0.	0.

NYLON 6,6 ISOTHERMAL WEIGHT LOSS 100MG.
RATE OF WT. LOSS IN PERCENT PER MINUTE

I.D.	IS25A	IS24A	IS27A
TEMP.	330	390	398
PERCENT WT.LOSS	RATE OF WT.LOSS	RATE OF WT.LOSS	RATE OF WT.LOSS
1	0.048	0.214	0.524
2	0.062	0.290	0.913
3	0.074	0.351	1.330
4	0.084	0.402	1.662
5	0.098	0.446	1.949
6	0.111	0.483	2.219
7	0.121	0.513	2.444
8	0.129	0.541	2.653
9	0.135	0.568	2.849
10	0.139	0.590	3.032
11	0.142	0.608	3.179
12	0.145	0.625	3.297
13	0.147	0.641	3.412
14	0.146	0.652	3.505
15	0.145	0.663	3.587
16	0.143	0.673	3.665
17	0.141	0.677	3.703
18	0.139	0.683	3.750
19	0.136	0.686	3.766
20	0.135	0.685	3.787
21	0.133	0.685	3.807
22	0.130	0.683	3.787
23	0.128	0.681	3.784
24	0.126	0.674	3.749
25	0.123	0.668	3.745
26	0.119	0.662	3.690
27	0.116	0.654	3.650
28	0.113	0.646	3.602
29	0.111	0.637	3.535
30	0.109	0.628	3.458
31	0.106	0.619	3.377
32	0.105	0.610	3.296
33	0.103	0.601	3.216
34	0.102	0.592	3.131
35	0.100	0.583	3.037
36	0.098	0.572	2.949
37	0.095	0.562	2.870
38	0.093	0.550	2.803
39	0.091	0.539	2.718
40	0.088	0.528	2.632
41	0.086	0.518	2.543

I.D.	IS25A	IS24A	IS27A
TEMP.	330	390	393
PERCENT WT.LOSS	RATE OF WT.LOSS	RATE OF WT.LOSS	RATE OF WT.LOSS
42	0.082	0.507	2.471
43	0.079	0.495	2.395
44	0.076	0.484	2.328
45	0.073	0.472	2.270
46	0.069	0.460	2.213
47	0.065	0.448	2.158
48	0.062	0.435	2.107
49	0.060	0.424	2.056
50	0.058	0.412	2.012
51	0.057	0.404	1.969
52	0.055	0.395	1.920
53	0.051	0.386	1.891
54	0.049	0.376	1.849
55	0.046	0.364	1.817
56	0.043	0.353	1.785
57	0.039	0.342	1.755
58	0.034	0.328	1.722
59	0.032	0.314	1.698
60	0.034	0.300	1.675
61	0.029	0.283	1.650
62	0.026	0.263	1.620
63	0.025	0.246	1.585
64	0.023	0.235	1.540
65	0.021	0.224	1.493
66	0.	0.210	1.456
67	0.	0.196	1.414
68	0.	0.184	1.360
69	0.	0.169	1.298
70	0.	0.158	1.234
71	0.	0.148	1.167
72	0.	0.138	1.100
73	0.	0.127	1.035
74	0.	0.115	0.958
75	0.	0.107	0.907
76	0.	0.096	0.858
77	0.	0.085	0.808
78	0.	0.073	0.734
79	0.	0.064	0.661
80	0.	0.	0.599
81	0.	0.	0.541
82	0.	0.	0.506
83	0.	0.	0.465
84	0.	0.	0.411
85	0.	0.	0.362
86	0.	0.	0.318
87	0.	0.	0.271
88	0.	0.	0.240
89	0.	0.	0.

NYLON 6,6 ISOTHERMAL WEIGHT LOSS 25CMG.
RATE OF WT. LOSS IN PERCENT PER MINUTE

I.D.	IS20A	IS19A
TEMP.	335	365
PERCENT WT.LOSS	RATE OF WT.LOSS	RATE OF WT.LOSS
1	0.073	0.431
2	0.056	0.467
3	0.080	0.546
4	0.097	0.601
5	0.112	0.648
6	0.125	0.697
7	0.136	0.721
8	0.142	0.744
9	0.145	0.775
10	0.147	0.780
11	0.151	0.786
12	0.152	0.802
13	0.154	0.793
14	0.154	0.809
15	0.154	0.786
16	0.154	0.795
17	0.153	0.762
18	0.150	0.759
19	0.148	0.743
20	0.146	0.709
21	0.143	0.660
22	0.141	0.611
23	0.140	0.571
24	0.140	0.546
25	0.138	0.532
26	0.136	0.526
27	0.135	0.524
28	0.133	0.526
29	0.130	0.528
30	0.128	0.527
31	0.126	0.525
32	0.121	0.521
33	0.117	0.515
34	0.115	0.507
35	0.112	0.497
36	0.107	0.485
37	0.096	0.470
38	0.083	0.455
39	0.084	0.440
40	0.087	0.424
41	0.	0.410
42	0.	0.396

I.D.	IS2CA	IS19A
TEMP.	335	355
PERCENT WT.LOSS	RATE OF WT.LOSS	RATE OF WT.LOSS
43	0.	0.383
44	0.	0.370
45	0.	0.360
46	0.	0.350
47	0.	0.342
48	0.	0.334
49	0.	0.326
50	0.	0.319
51	0.	0.311
52	0.	0.303
53	0.	0.296
54	0.	0.287
55	0.	0.279
56	0.	0.269
57	0.	0.260
58	0.	0.250
59	0.	0.239
60	0.	0.230
61	0.	0.221
62	0.	0.213
63	0.	0.203
64	0.	0.193
65	0.	0.181
66	0.	0.171
67	0.	0.161
68	0.	0.151
69	0.	0.141
70	0.	0.130
71	0.	0.120
72	0.	0.109
73	0.	0.099
74	0.	0.090
75	0.	0.083
76	0.	0.073
77	0.	0.068
78	0.	0.061
79	0.	0.055
80	0.	0.048
81	0.	0.041
82	0.	0.027
83	0.	0.

NYLON 6,6 ISOTHERMAL WEIGHT LOSS 50MG.
RATE OF WT. LOSS IN PERCENT PER MINUTE

ISOC. TEMP.	IS30A 374	IS28A 395
PERCENT WT.LOSS	RATE OF WT.LOSS	RATE OF WT.LOSS
1	0.390	0.272
2	0.579	0.624
3	0.732	1.046
4	0.857	1.397
5	0.952	1.671
6	1.037	1.901
7	1.110	2.100
8	1.175	2.294
9	1.233	2.445
10	1.280	2.586
11	1.316	2.711
12	1.340	2.816
13	1.360	2.911
14	1.373	2.999
15	1.381	3.071
16	1.385	3.123
17	1.379	3.159
18	1.371	3.170
19	1.358	3.191
20	1.345	3.222
21	1.321	3.235
22	1.302	3.244
23	1.274	3.257
24	1.245	3.255
25	1.218	3.238
26	1.186	3.234
27	1.151	3.216
28	1.118	3.195
29	1.090	3.161
30	1.063	3.137
31	1.039	3.098
32	1.010	3.053
33	0.997	3.012
34	0.980	2.967
35	0.960	2.925
36	0.947	2.878
37	0.933	2.830
38	0.924	2.796
39	0.915	2.751
40	0.909	2.702
41	0.896	2.637
42	0.887	2.586
43	0.878	2.535

I.D.	IS30A	IS28A
TEMP.	374	395
PERCENT WT.LOSS	RATE OF WT.LOSS	RATE OF WT.LOSS
44	0.867	2.476
45	0.862	2.421
46	0.855	2.355
47	0.850	2.306
48	0.834	2.249
49	0.815	2.192
50	0.798	2.132
51	0.775	2.092
52	0.753	2.034
53	0.740	1.968
54	0.725	1.913
55	0.705	1.867
56	0.684	1.805
57	0.666	1.748
58	0.651	1.697
59	0.634	1.650
60	0.615	1.587
61	0.592	1.543
62	0.564	1.496
63	0.545	1.445
64	0.517	1.412
65	0.492	1.358
66	0.469	1.299
67	0.455	1.238
68	0.434	1.178
69	0.427	1.122
70	0.418	1.068
71	0.415	1.037
72	0.388	0.995
73	0.372	0.943
74	0.339	0.889
75	0.320	0.844
76	0.305	0.795
77	0.268	0.726
78	0.248	0.680
79	0.232	0.623
80	0.	0.590
81	0.	0.558
82	0.	0.484
83	0.	0.428
84	0.	0.399
85	0.	0.367
86	0.	0.317
87	0.	0.293
88	0.	0.283
89	0.	0.170
90	0.	0.187
91	0.	0.122
92	0.	0.

NYLON 6,6 ISOTHERMAL 100MG. (NEW BAL.)
RATE OF WT. LOSS IN PERCENT PER MINUTE

I.D.	IS5CA	IS51A	IS52A	IS54A	IS53A	IS55A
TEMP.	346	355	371	371	380	387
PERCENT	RATE OF	RATE OF	RATE OF	RATE OF	RATE OF	RATE OF
WT.LOSS	WT.LOSS	WT.LOSS	WT.LOSS	WT.LOSS	WT.LOSS	WT.LOSS
1	0.075	0.068	0.130	0.149	0.173	0.065
2	0.073	0.091	0.160	0.184	0.273	0.159
3	0.085	0.128	0.220	0.262	0.393	0.269
4	0.121	0.142	0.335	0.347	0.533	0.423
5	0.149	0.191	0.427	0.416	0.651	0.605
6	0.170	0.241	0.499	0.480	0.757	0.782
7	0.186	0.281	0.557	0.545	0.840	0.912
8	0.198	0.311	0.606	0.595	0.906	1.018
9	0.209	0.329	0.644	0.631	0.956	1.097
10	0.219	0.336	0.672	0.650	0.997	1.154
11	0.230	0.338	0.696	0.657	1.024	1.189
12	0.241	0.343	0.713	0.659	1.047	1.219
13	0.249	0.349	0.727	0.664	1.093	1.241
14	0.240	0.356	0.741	0.676	1.109	1.258
15	0.239	0.360	0.753	0.689	1.134	1.279
16	0.247	0.365	0.765	0.709	1.154	1.306
17	0.258	0.373	0.776	0.733	1.170	1.347
18	0.272	0.381	0.784	0.750	1.181	1.381
19	0.272	0.389	0.791	0.762	1.186	1.415
20	0.280	0.396	0.793	0.768	1.187	1.442
21	0.280	0.401	0.793	0.766	1.182	1.460
22	0.283	0.405	0.791	0.760	1.174	1.466
23	0.278	0.408	0.789	0.748	1.166	1.462
24	0.287	0.409	0.785	0.736	1.157	1.455
25	0.231	0.408	0.781	0.715	1.146	1.435
26	0.221	0.406	0.777	0.692	1.132	1.411
27	0.213	0.403	0.773	0.671	1.120	1.389
28	0.208	0.398	0.768	0.650	1.111	1.362
29	0.204	0.392	0.762	0.632	1.103	1.338
30	0.201	0.383	0.757	0.621	1.096	1.322
31	0.197	0.373	0.751	0.618	1.090	1.312
32	0.194	0.361	0.744	0.627	1.084	1.306
33	0.190	0.348	0.738	0.642	1.080	1.305
34	0.187	0.335	0.730	0.652	1.075	1.304
35	0.184	0.325	0.721	0.655	1.069	1.305
36	0.181	0.316	0.712	0.654	1.064	1.305
37	0.177	0.309	0.702	0.649	1.058	1.307
38	0.174	0.303	0.692	0.635	1.054	1.307
39	0.171	0.298	0.680	0.619	1.047	1.306
40	0.167	0.292	0.668	0.606	1.038	1.300
41	0.163	0.287	0.654	0.594	1.028	1.292
42	0.159	0.283	0.641	0.588	1.013	1.280

I.C. TEMP. PERCENT WT.LOSS	IS50A 346 RATE OF WT.LOSS	IS51A 355 RATE OF WT.LOSS	IS52A 371 RATE OF WT.LOSS	IS54A 371 RATE OF WT.LOSS	IS53A 380 RATE OF WT.LOSS	IS55A 387 RATE OF WT.LOSS
43	0.155	0.279	0.626	0.590	0.995	1.267
44	0.151	0.276	0.611	0.581	0.971	1.250
45	0.147	0.272	0.593	0.571	0.944	1.229
46	0.142	0.267	0.575	0.553	0.915	1.201
47	0.137	0.262	0.556	0.530	0.886	1.168
48	0.133	0.256	0.537	0.515	0.855	1.130
49	0.130	0.250	0.517	0.510	0.826	1.086
50	0.126	0.243	0.496	0.500	0.800	1.038
51	0.121	0.235	0.474	0.477	0.774	0.987
52	0.112	0.227	0.453	0.431	0.751	0.937
53	0.108	0.220	0.436	0.408	0.729	0.888
54	0.104	0.213	0.419	0.401	0.713	0.850
55	0.100	0.205	0.402	0.393	0.696	0.824
56	0.095	0.196	0.387	0.384	0.677	0.805
57	0.090	0.189	0.372	0.374	0.666	0.802
58	0.085	0.185	0.360	0.363	0.659	0.799
59	0.079	0.180	0.349	0.343	0.649	0.798
60	0.073	0.170	0.339	0.308	0.636	0.794
61	0.068	0.152	0.329	0.290	0.613	0.785
62	0.064	0.139	0.318	0.287	0.580	0.772
63	0.062	0.131	0.306	0.268	0.551	0.745
64	0.058	0.123	0.296	0.256	0.524	0.721
65	0.053	0.114	0.284	0.250	0.494	0.700
66	0.048	0.105	0.271	0.245	0.458	0.675
67	0.043	0.098	0.257	0.239	0.423	0.645
68	0.040	0.088	0.238	0.232	0.391	0.606
69	0.036	0.081	0.218	0.223	0.360	0.559
70	0.033	0.073	0.195	0.196	0.331	0.509
71	0.027	0.063	0.174	0.192	0.313	0.465
72	0.024	0.058	0.159	0.179	0.303	0.436
73	0.	0.	0.151	0.162	0.289	0.411
74	0.	0.	0.146	0.146	0.279	0.388
75	0.	0.	0.141	0.135	0.266	0.368
76	0.	0.	0.133	0.124	0.250	0.348
77	0.	0.	0.122	0.113	0.231	0.326
78	0.	0.	0.113	0.100	0.207	0.307
79	0.	0.	0.097	0.	0.187	0.290
80	0.	0.	0.082	0.	0.166	0.268
81	0.	0.	0.070	0.	0.145	0.243
82	0.	0.	0.	0.	0.	0.221
83	0.	0.	0.	0.	0.	0.201
84	0.	0.	0.	0.	0.	0.

NYLON 6,10 250 MG.
RATE OF WT. LOSS IN PERCENT PER MINUTE

I.D.	ISO2B	ISO5B	ISO3B	ISO8B	ISO6B	ISO7B
TEMP.	370	371	398	410	413	432
PERCENT WT.LOSS	RATE OF WT.LOSS	RATE OF WT.LOSS	RATE OF WT.LOSS	RATE OF WT.LOSS	RATE OF WT.LOSS	RATE OF WT.LOSS
1	0.034	0.079	0.275	0.501	0.460	0.835
2	0.051	0.103	0.414	0.710	0.876	1.422
3	0.050	0.119	0.621	0.876	1.185	1.973
4	0.056	0.131	0.774	1.022	1.441	2.509
5	0.059	0.136	0.883	1.154	1.663	3.014
6	0.058	0.138	0.982	1.268	1.840	3.497
7	0.062	0.146	1.064	1.367	1.995	3.949
8	0.066	0.154	1.130	1.459	2.126	4.367
9	0.068	0.162	1.181	1.535	2.235	4.762
10	0.070	0.169	1.223	1.615	2.326	5.107
11	0.071	0.176	1.254	1.676	2.403	5.418
12	0.071	0.182	1.249	1.730	2.476	5.696
13	0.071	0.187	1.252	1.782	2.534	5.944
14	0.072	0.192	1.245	1.830	2.570	6.169
15	0.072	0.196	1.226	1.872	2.597	6.377
16	0.073	0.200	1.192	1.904	2.606	6.536
17	0.073	0.203	1.151	1.935	2.614	6.685
18	0.073	0.205	1.111	1.963	2.608	6.794
19	0.073	0.207	1.063	1.969	2.591	6.905
20	0.073	0.209	1.025	1.985	2.563	7.010
21	0.071	0.210	0.991	1.979	2.536	7.047
22	0.070	0.210	0.970	1.984	2.497	7.095
23	0.069	0.209	0.956	1.968	2.460	7.140
24	0.068	0.209	0.944	1.965	2.418	7.121
25	0.067	0.208	0.941	1.962	2.367	7.118
26	0.066	0.207	0.938	1.933	2.320	7.102
27	0.064	0.204	0.936	1.918	2.270	7.042
28	0.062	0.202	0.931	1.886	2.225	6.986
29	0.061	0.199	0.929	1.866	2.177	6.920
30	0.060	0.195	0.925	1.844	2.130	6.844
31	0.060	0.192	0.921	1.830	2.097	6.753
32	0.062	0.189	0.912	1.804	2.054	6.649
33	0.058	0.185	0.909	1.777	2.015	6.537
34	0.055	0.182	0.903	1.752	1.969	6.424
35	0.047	0.180	0.897	1.725	1.930	6.300
36	0.049	0.177	0.889	1.719	1.882	6.170
37	0.054	0.174	0.881	1.703	1.825	6.034
38	0.052	0.172	0.871	1.685	1.769	5.902
39	0.050	0.169	0.862	1.677	1.715	5.766
40	0.049	0.167	0.857	1.675	1.665	5.621
41	0.048	0.163	0.851	1.671	1.624	5.445
42	0.047	0.159	0.843	1.666	1.580	5.276
43	0.045	0.155	0.834	1.660	1.555	5.091
44	0.044	0.151	0.823	1.648	1.524	4.921
45	0.043	0.147	0.811	1.645	1.499	4.751
46	0.042	0.144	0.800	1.641	1.498	4.603

I.D.	ISO2B	ISO5B	ISO3B	ISO8B	ISO6B	ISO7B
TEMP.	370	371	398	410	413	432
PERCENT WT.LOSS	RATE OF WT.LOSS	RATE OF WT.LOSS	RATE OF WT.LOSS	RATE OF WT.LOSS	RATE OF WT.LOSS	RATE OF WT.LOSS
47	0.041	0.141	0.790	1.622	1.489	4.435
48	0.040	0.139	0.780	1.606	1.491	4.310
49	0.039	0.136	0.767	1.584	1.487	4.180
50	0.037	0.134	0.756	1.562	1.490	4.059
51	0.034	0.132	0.742	1.537	1.479	3.971
52	0.033	0.130	0.725	1.507	1.483	3.869
53	0.031	0.127	0.713	1.475	1.480	3.757
54	0.029	0.125	0.696	1.444	1.482	3.666
55	0.027	0.123	0.682	1.414	1.477	3.566
56	0.025	0.120	0.665	1.381	1.471	3.494
57	0.024	0.117	0.653	1.352	1.457	3.410
58	0.	0.113	0.635	1.320	1.432	3.336
59	0.	0.110	0.613	1.287	1.409	3.274
60	0.	0.106	0.594	1.253	1.380	3.212
61	0.	0.102	0.575	1.218	1.363	3.165
62	0.	0.099	0.556	1.183	1.331	3.129
63	0.	0.096	0.540	1.153	1.287	3.090
64	0.	0.093	0.524	1.120	1.239	3.054
65	0.	0.088	0.509	1.085	1.188	3.014
66	0.	0.084	0.496	1.053	1.145	2.976
67	0.	0.081	0.478	1.026	1.102	2.935
68	0.	0.077	0.459	0.996	1.061	2.902
69	0.	0.074	0.438	0.964	1.024	2.871
70	0.	0.070	0.416	0.933	0.994	2.822
71	0.	0.065	0.397	0.900	0.961	2.782
72	0.	0.059	0.378	0.870	0.923	2.744
73	0.	0.053	0.358	0.833	0.884	2.704
74	0.	0.048	0.348	0.803	0.851	2.641
75	0.	0.045	0.337	0.770	0.805	2.564
76	0.	0.044	0.322	0.740	0.774	2.487
77	0.	0.044	0.310	0.712	0.745	2.402
78	0.	0.044	0.296	0.680	0.711	2.314
79	0.	0.039	0.283	0.645	0.679	2.198
80	0.	0.036	0.267	0.614	0.646	2.074
81	0.	0.034	0.254	0.581	0.589	1.940
82	0.	0.032	0.238	0.547	0.541	1.802
83	0.	0.030	0.217	0.513	0.499	1.625
84	0.	0.029	0.202	0.484	0.466	1.435
85	0.	0.026	0.193	0.446	0.438	1.287
86	0.	0.024	0.174	0.410	0.408	1.155
87	0.	0.022	0.151	0.373	0.373	1.058
88	0.	0.018	0.128	0.342	0.335	0.931
89	0.	0.016	0.120	0.309	0.306	0.822
90	0.	0.	0.109	0.279	0.269	0.724
91	0.	0.	0.	0.241	0.237	0.653
92	0.	0.	0.	0.210	0.202	0.588
93	0.	0.	0.	0.178	0.	0.490
94	0.	0.	0.	0.146	0.	0.389
95	0.	0.	0.	0.114	0.	0.
96	0.	0.	0.	0.091	0.	0.
97	0.	0.	0.	0.	0.	0.

NYLCN 6,10 100 MG.
RATE CF WT. LCSS IN PERCENT PER MINUTE

IJC.	IS16B	IS13B	ISC1B
TEMP.	335	378	395
PERCENT WT.LCSS	RATE CF WT.LCSS	RATE CF WT.LOSS	RATE CF WT.LOSS
1	C.036	0.872	0.555
2	C.038	0.920	0.743
3	0.032	1.323	0.882
4	C.038	1.743	0.991
5	C.045	2.095	1.076
6	C.050	2.401	1.141
7	C.053	2.656	1.189
8	C.057	2.875	1.223
9	C.060	3.141	1.245
10	C.063	3.305	1.229
11	C.066	3.526	1.220
12	C.068	3.655	1.199
13	0.069	3.803	1.184
14	C.069	3.988	1.134
15	C.070	4.092	1.092
16	C.071	4.194	1.043
17	C.071	4.294	1.002
18	C.071	4.379	0.955
19	C.071	4.445	0.936
20	C.071	4.509	0.927
21	C.070	4.568	0.910
22	C.070	4.601	0.910
23	C.069	4.634	0.912
24	C.069	4.691	0.909
25	C.068	4.702	0.914
26	C.068	4.755	0.914
27	C.066	4.743	0.912
28	C.066	4.796	0.907
29	C.064	4.763	0.901
30	C.063	4.731	0.897
31	C.063	4.755	0.894
32	C.062	4.708	0.885
33	C.061	4.744	0.881
34	C.060	4.682	0.877
35	C.059	4.650	0.870
36	C.058	4.583	0.868
37	C.056	4.516	0.866
38	C.056	4.402	0.866
39	C.056	4.330	0.868
40	C.054	4.272	0.867
41	C.054	4.212	0.863
42	C.053	4.153	0.856
43	C.051	4.091	0.853
44	C.050	4.032	0.842
45	C.049	3.971	0.815

I.D.	IS16B	IS13B	IS01B
TEMP.	335	278	395
PERCENT	RATE OF	RATE OF	RATE OF
WT.LOSS	WT.LOSS	WT.LOSS	WT.LOSS
46	C.048	3.904	0.792
47	C.048	3.834	C.782
48	C.046	3.77C	0.771
49	C.044	3.703	0.750
5C	C.042	3.635	0.76C
51	C.040	3.563	0.748
52	C.038	3.490	0.748
53	C.035	3.421	0.746
54	0.034	3.361	0.741
55	C.034	3.276	0.735
56	C.033	3.199	0.709
57	C.030	3.109	0.651
58	C.025	3.02C	0.640
59	C.	2.916	0.620
6C	C.	2.337	0.597
61	0.	2.746	0.575
62	C.	2.653	0.558
63	C.	2.557	0.539
64	C.	2.461	0.525
65	C.	2.371	0.501
66	C.	2.275	0.479
67	C.	2.174	0.461
68	C.	2.072	0.446
69	C.	1.98C	0.427
7C	C.	1.889	0.409
71	C.	1.798	0.395
72	C.	1.698	0.372
73	C.	1.600	0.356
74	C.	1.499	0.338
75	C.	1.405	0.322
76	C.	1.307	0.298
77	C.	1.214	0.285
78	C.	1.141	0.251
79	C.	1.064	0.188
8C	C.	0.997	0.181
81	C.	0.934	0.200
82	C.	0.857	0.190
83	C.	0.785	0.183
84	C.	0.714	0.169
85	C.	0.641	0.151
86	C.	0.581	0.
87	C.	0.514	0.
88	C.	0.451	0.
89	C.	0.389	0.
9C	C.	0.342	0.
91	C.	0.310	0.
92	C.	0.243	0.
93	C.	0.14C	0.
94	C.	0.	0.

NYLON 6,10 100 MG. (NEW BAL.)
RATE OF WT. LOSS IN PERCENT PER MINUTE

I.D.	IS54B	IS51B	IS53B	IS50B	IS52B
TEMP.	375	380	392	400	404
PERCENT WT.LOSS	RATE OF WT.LOSS	RATE OF WT.LOSS	RATE OF WT.LOSS	RATE OF WT.LOSS	RATE OF WT.LOSS
1	0.107	0.164	0.149	0.128	0.199
2	0.160	0.201	0.294	0.255	0.386
3	0.181	0.199	0.359	0.423	0.518
4	0.196	0.189	0.378	0.539	0.631
5	0.206	0.197	0.381	0.603	0.736
6	0.213	0.220	0.407	0.621	0.815
7	0.223	0.243	0.445	0.596	0.860
8	0.237	0.264	0.471	0.546	0.890
9	0.245	0.283	0.479	0.504	0.902
10	0.250	0.300	0.489	0.494	0.898
11	0.259	0.319	0.520	0.526	0.885
12	0.275	0.337	0.569	0.571	0.872
13	0.294	0.351	0.612	0.607	0.873
14	0.304	0.364	0.644	0.635	0.902
15	0.306	0.374	0.670	0.656	0.940
16	0.308	0.381	0.694	0.672	0.980
17	0.312	0.385	0.712	0.704	1.023
18	0.315	0.387	0.722	0.737	1.067
19	0.313	0.388	0.732	0.767	1.110
20	0.306	0.388	0.734	0.790	1.147
21	0.295	0.385	0.730	0.810	1.177
22	0.293	0.379	0.721	0.828	1.199
23	0.306	0.373	0.712	0.842	1.216
24	0.323	0.367	0.697	0.853	1.226
25	0.334	0.361	0.681	0.856	1.230
26	0.339	0.360	0.674	0.852	1.225
27	0.337	0.361	0.675	0.838	1.218
28	0.331	0.362	0.669	0.822	1.208
29	0.322	0.364	0.672	0.807	1.197
30	0.315	0.367	0.679	0.810	1.190
31	0.311	0.370	0.690	0.832	1.189
32	0.310	0.371	0.703	0.875	1.189
33	0.313	0.371	0.720	0.918	1.196
34	0.318	0.371	0.725	0.952	1.204
35	0.317	0.366	0.716	0.973	1.213
36	0.313	0.360	0.697	0.981	1.221
37	0.304	0.355	0.670	0.978	1.222
38	0.288	0.351	0.634	0.967	1.220
39	0.268	0.347	0.601	0.950	1.220
40	0.249	0.342	0.581	0.931	1.218
41	0.241	0.340	0.578	0.916	1.214
42	0.241	0.339	0.583	0.906	1.207
43	0.246	0.340	0.597	0.901	1.200
44	0.244	0.340	0.601	0.900	1.188
45	0.240	0.337	0.603	0.900	1.175

I.D.	IS54B	IS51B	IS53B	IS50B	IS52B
TEMP.	376	380	392	400	404
PERCENT WT.LOSS	RATE OF WT.LOSS	RATE OF WT.LOSS	RATE OF WT.LOSS	RATE OF WT.LOSS	RATE OF WT.LOSS
46	C.235	0.333	0.600	0.897	1.163
47	C.230	0.325	0.593	0.891	1.137
48	C.225	0.315	0.582	0.877	1.112
49	C.221	0.300	0.571	0.858	1.089
50	C.219	0.285	0.553	0.838	1.068
51	C.215	0.267	0.528	0.814	1.052
52	C.209	0.255	0.497	0.792	1.039
53	C.202	0.248	0.467	0.772	1.029
54	C.197	0.244	0.452	0.755	1.019
55	C.191	0.244	0.446	0.740	1.008
56	C.186	0.248	0.452	0.729	0.993
57	C.180	0.249	0.460	0.719	0.974
58	C.174	0.246	0.460	0.711	0.950
59	C.168	0.247	0.429	0.702	0.920
60	C.157	0.245	0.382	0.694	0.887
61	C.147	0.237	0.361	0.692	0.855
62	C.150	0.223	0.380	0.690	0.822
63	C.145	0.209	0.382	0.684	0.796
64	C.139	0.199	0.375	0.673	0.781
65	C.135	0.188	0.361	0.656	0.755
66	C.129	0.176	0.345	0.627	0.735
67	C.122	0.165	0.329	0.601	0.718
68	C.118	0.156	0.314	0.575	0.701
69	C.115	0.148	0.300	0.543	0.683
70	C.110	0.143	0.282	0.510	0.668
71	C.101	0.142	0.277	0.477	0.652
72	C.101	0.140	0.277	0.446	0.639
73	C.095	0.135	0.265	0.423	0.632
74	C.092	0.130	0.254	0.409	0.622
75	0.	0.124	0.242	0.402	0.602
76	0.	0.112	0.229	0.392	0.578
77	C.	0.102	0.217	0.383	0.551
78	C.	0.095	0.209	0.370	0.524
79	C.	0.084	0.198	0.354	0.492
80	C.	0.081	0.174	0.337	0.458
81	C.	0.076	0.176	0.319	0.425
82	C.	0.074	0.167	0.301	0.410
83	C.	0.070	0.157	0.285	0.397
84	C.	0.066	0.147	0.265	0.374
85	C.	0.	0.137	0.242	0.350
86	C.	0.	0.128	0.224	0.325
87	C.	0.	0.	0.205	0.298
88	C.	0.	0.	0.188	0.268
89	C.	0.	0.	0.169	0.232
90	C.	C.	0.	0.148	0.199
91	C.	0.	0.	0.131	0.166
92	C.	0.	0.	0.116	0.159
93	C.	0.	0.	0.	0.132
94	C.	0.	0.	0.	0.109
95	C.	0.	0.	0.	0.086
96	C.	0.	0.	0.	0.

NYLON 6,10 ISOTHERMAL 500MG.
RATE OF WT. LOSS IN PERCENT PER MINUTE

ISO. TEMP.	ISO18 376	ISO19 387
PERCENT WT.LOSS	RATE OF WT.LOSS	RATE OF WT.LOSS
1	0.575	0.126
2	0.552	0.183
3	0.798	0.238
4	0.864	0.288
5	1.619	0.332
6	1.779	0.367
7	0.779	0.396
8	0.821	0.418
9	0.852	0.436
10	0.886	0.447
11	0.907	0.457
12	0.931	0.468
13	0.945	0.478
14	0.961	0.487
15	0.971	0.496
16	0.981	0.503
17	0.993	0.509
18	0.996	0.512
19	1.004	0.515
20	1.004	0.516
21	1.009	0.515
22	1.007	0.514
23	1.007	0.513
24	1.003	0.510
25	1.001	0.507
26	0.996	0.504
27	0.991	0.501
28	0.986	0.498
29	0.979	0.494
30	0.973	0.489
31	0.965	0.485
32	0.959	0.481
33	0.949	0.476
34	0.940	0.469
35	0.929	0.462
36	0.919	0.456
37	0.908	0.448
38	0.896	0.439
39	0.885	0.432
40	0.873	0.425
41	0.858	0.418
42	0.844	0.412
43	0.829	0.407
44	0.813	0.401
45	0.797	0.395

I.D.	IS018	IS019
TEMP.	376	387
PERCENT	RATE OF	RATE OF
WT.LOSS	WT.LOSS	WT.LOSS
46	C.781	0.392
47	C.764	0.387
48	C.746	0.381
49	C.729	0.378
50	C.711	0.373
51	C.694	0.366
52	C.677	0.361
53	C.659	0.352
54	C.641	0.341
55	C.624	0.331
56	C.606	0.319
57	C.590	0.310
58	C.571	0.300
59	C.554	0.291
60	C.536	0.285
61	C.518	0.277
62	C.501	0.269
63	C.485	0.259
64	C.467	0.250
65	C.450	0.241
66	C.434	0.233
67	C.417	0.224
68	C.400	0.218
69	C.384	0.213
70	C.370	0.208
71	C.355	0.206
72	C.341	0.204
73	C.326	0.201
74	C.310	0.194
75	C.294	0.185
76	C.276	0.174
77	C.259	0.164
78	C.241	0.157
79	C.227	0.150
80	C.212	0.144
81	C.199	0.136
82	C.185	0.128
83	C.172	0.
84	C.160	0.
85	C.145	0.
86	C.128	0.
87	C.112	0.
88	C.107	0.
89	C.098	0.
90	C.090	0.
91	C.082	0.
92	C.073	0.
93	C.064	0.
94	C.054	0.
95	C.	0.

APPENDIX II

PROGRAMMED TEMPERATURE NYLON 6.6 AND 6.10 RATE OF WEIGHT LOSS DATA

NYLON 6,10 100MG. SAMPLES
H.R. = HEATING RATE IN DEGREES C PER HOUR
RATE OF WT. LOSS IN PERCENT PER MINUTE

I.C./H.R.	01P/270		02P/440		03P/ 78		04P/ 75		05P/150	
PERCENT WT.LOSS	RATE OF WT.LOSS	TEMP. DEG C	RATE OF WT.LOSS	TEMP. DEG C	RATE OF WT.LOSS	TEMP. DEG C	RATE OF WT.LOSS	TEMP. DEG C	RATE OF WT.LOSS	TEMP. DEG C
1	0.116	175.8	0.274	324.9	0.048	343.4	0.051	323.6	0.062	331.0
2	0.	299.5	0.281	373.4	0.108	362.1	0.079	362.0	0.173	358.4
3	0.073	350.3	0.589	384.0	0.224	371.8	0.638	368.6	0.264	372.9
4	0.288	374.8	0.637	401.0	0.282	376.5	0.619	371.1	0.453	381.7
5	0.772	390.0	1.828	408.8	0.353	381.1	0.769	372.6	0.642	386.9
6	0.807	396.7	2.642	412.2	0.425	385.0	0.666	375.0	0.914	391.2
7	1.289	401.2	3.341	414.9	0.550	387.9	0.545	377.7	1.099	393.6
8	1.836	405.1	4.058	417.0	0.701	390.3	0.468	381.4	1.338	396.0
9	2.396	407.3	4.573	418.8	0.823	392.1	0.461	386.2	1.507	397.7
10	2.743	409.0	5.030	420.5	0.920	393.6	0.603	389.4	1.718	399.4
11	3.132	410.6	5.511	421.9	1.037	395.1	0.758	391.9	1.893	400.9
12	3.354	412.0	6.016	423.3	1.107	396.3	0.874	393.9	2.054	402.1
13	3.834	413.5	6.474	424.5	1.196	397.4	1.001	395.5	2.225	403.2
14	4.118	414.8	6.975	425.7	1.306	398.5	1.114	397.0	2.369	404.2
15	4.733	415.9	7.395	426.7	1.383	399.4	1.214	398.3	2.504	405.2
16	5.237	416.8	7.996	427.7	1.453	400.4	1.319	399.5	2.698	406.2
17	5.215	417.6	8.570	428.7	1.516	401.3	1.402	400.5	2.840	407.1
18	5.771	418.6	9.193	429.6	1.581	402.1	1.524	401.5	3.052	407.9
19	6.123	419.3	9.734	430.4	1.642	402.9	1.607	402.4	3.189	408.6
20	6.118	420.1	9.841	431.1	1.743	403.7	1.685	403.3	3.301	409.4
21	6.593	420.8	10.309	431.9	1.813	404.5	1.753	404.2	3.393	410.1
22	6.804	421.5	10.549	432.6	1.889	405.3	1.827	404.9	3.529	410.8
23	7.009	422.2	10.945	433.3	1.963	405.9	1.908	405.7	3.697	411.4
24	6.972	423.0	11.571	433.9	2.017	406.6	1.981	406.4	3.829	412.0
25	7.366	423.6	12.056	434.6	2.086	407.3	2.052	407.1	3.957	412.6
26	7.715	424.2	12.391	435.2	2.153	407.9	2.143	407.8	4.035	413.2
27	7.785	424.7	12.749	435.8	2.271	408.5	2.218	408.5	4.160	413.8
28	8.056	425.5	13.093	436.4	2.342	409.1	2.291	409.1	4.281	414.4
29	8.525	426.0	13.286	436.9	2.383	409.6	2.343	409.6	4.399	414.9
30	8.876	426.5	13.589	437.5	2.465	410.1	2.406	410.2	4.454	415.3
31	9.213	427.1	13.790	438.0	2.520	410.7	2.406	410.7	4.540	415.9
32	9.257	427.5	14.138	438.6	2.574	411.2	2.459	411.2	4.625	416.4
33	9.385	428.0	14.478	439.1	2.604	411.7	2.501	411.7	4.708	416.9
34	9.541	428.5	14.811	439.6	2.658	412.2	2.539	412.3	4.756	417.3
35	9.694	429.0	14.850	440.1	2.710	412.7	2.576	412.8	4.815	417.8
36	9.899	429.4	15.125	440.6	2.762	413.2	2.633	413.2	4.849	418.2
37	9.828	429.8	15.277	441.0	2.812	413.6	2.661	413.7	4.899	418.7
38	9.902	430.4	15.503	441.5	2.949	414.1	2.689	414.2	4.949	419.2
39	9.974	430.8	16.058	441.9	3.006	414.5	2.756	414.7	5.034	419.7
40	10.314	431.3	16.175	442.4	3.062	415.0	2.783	415.1	5.085	420.1
41	10.399	431.7	16.244	442.8	3.117	415.4	2.809	415.6	5.135	420.6
42	10.354	432.1	16.328	443.3	3.197	415.8	2.810	416.0	5.151	421.0
43	10.315	432.6	16.412	443.7	3.256	416.2	2.834	416.5	5.194	421.4
44	10.329	433.1	16.408	444.2	3.274	416.7	2.858	416.9	5.236	421.9
45	10.571	433.5	16.541	444.7	3.334	417.0	2.880	417.3	5.321	422.3
46	10.746	434.0	16.674	445.1	3.393	417.4	2.896	417.7	5.343	422.7
47	10.918	434.4	16.623	445.5	3.451	417.8	2.892	418.2	5.365	423.1
48	11.205	434.8	16.644	445.9	3.508	418.2	2.912	418.6	5.387	423.6
49	11.209	435.2	16.665	446.4	3.420	418.5	2.932	419.0	5.390	424.0
50	11.361	435.6	16.794	446.8	3.451	418.9	2.951	419.4	5.410	424.4

PERCENT WT.LOSS	RATE OF WT.LOSS	TEMP. DEG C	RATE OF WT.LOSS	TEMP. DEG C	RATE OF WT.LOSS	TEMP. DEG C	RATE OF WT.LOSS	TEMP. DEG C	RATE OF WT.LOSS	TEMP. DEG C
51	11.411	436.0	16.780	447.2	3.482	419.3	2.976	419.9	5.430	424.8
52	11.463	436.4	16.765	447.6	3.513	419.6	3.002	420.3	5.390	425.3
53	11.514	436.8	16.900	448.0	3.544	420.0	3.029	420.7	5.406	425.7
54	11.366	437.1	16.834	448.5	3.574	420.4	3.017	421.0	5.423	426.1
55	11.304	437.6	16.767	448.9	3.483	420.7	3.021	421.4	5.439	426.5
56	11.375	438.0	16.717	449.3	3.612	421.0	3.073	421.7	5.459	426.9
57	11.305	438.3	16.653	449.8	3.585	421.3	3.062	422.1	5.450	427.3
58	11.275	438.8	16.588	450.2	3.558	421.7	3.052	422.5	5.440	427.7
59	11.682	439.1	16.524	450.7	3.531	422.1	3.042	422.9	5.431	428.1
60	11.600	439.5	16.613	451.1	3.504	422.5	3.049	423.3	5.443	428.5
61	11.276	439.9	16.549	451.5	3.476	422.8	3.042	423.6	5.411	428.9
62	11.105	440.3	16.484	452.0	3.517	423.1	3.035	424.0	5.379	429.4
63	11.039	440.7	16.271	452.4	3.470	423.5	3.014	424.4	5.347	429.8
64	10.972	441.1	16.186	452.8	3.423	423.9	3.004	424.8	5.316	430.2
65	10.807	441.5	16.099	453.3	3.375	424.3	2.995	425.1	5.266	430.6
66	10.684	441.9	16.013	453.7	3.304	424.7	2.972	425.5	5.215	431.1
67	10.559	442.4	15.926	454.2	3.267	425.1	2.949	425.8	5.209	431.5
68	10.635	442.8	15.664	454.6	3.229	425.5	2.927	426.2	5.154	431.9
69	10.582	443.2	15.552	455.1	3.191	425.9	2.904	426.6	5.098	432.4
70	10.407	443.6	15.440	455.5	3.157	426.3	2.881	427.0	5.041	432.8
71	10.328	444.1	15.274	456.0	3.123	426.8	2.880	427.3	4.962	433.3
72	9.898	444.5	15.106	456.4	3.089	427.2	2.846	427.7	4.898	433.8
73	9.906	444.9	14.936	456.9	3.046	427.6	2.811	428.1	4.834	434.2
74	9.725	445.4	14.712	457.4	3.017	428.1	2.776	428.5	4.769	434.7
75	9.427	445.9	14.520	457.9	2.988	428.5	2.732	428.9	4.703	435.2
76	9.365	446.4	14.325	458.3	2.869	428.9	2.688	429.3	4.659	435.7
77	9.522	446.8	14.206	458.8	2.825	429.4	2.644	429.7	4.584	436.2
78	9.268	447.2	13.898	459.3	2.765	429.8	2.598	430.1	4.508	436.7
79	9.056	447.7	13.535	459.8	2.708	430.2	2.546	430.5	4.420	437.2
80	8.794	448.2	13.185	460.3	2.614	430.7	2.495	430.9	4.299	437.7
81	8.376	448.7	12.825	460.8	2.557	431.3	2.419	431.4	4.200	438.2
82	7.937	449.2	12.535	461.4	2.465	431.8	2.363	431.8	4.099	438.8
83	7.608	449.8	12.063	462.0	2.400	432.3	2.325	432.2	3.989	439.3
84	7.363	450.5	11.681	462.6	2.333	432.9	2.243	432.7	3.864	439.9
85	7.027	451.1	11.286	463.2	2.246	433.4	2.141	433.2	3.778	440.5
86	6.720	451.7	10.780	463.8	2.155	434.0	2.050	433.7	3.634	441.1
87	6.328	452.4	10.345	464.4	2.042	434.6	1.933	434.2	3.485	441.8
88	5.975	453.1	9.892	465.1	1.934	435.3	1.842	434.8	3.368	442.4
89	5.334	453.8	9.526	465.8	1.830	436.0	1.769	435.4	3.178	443.1
90	4.897	454.7	8.999	466.6	1.707	436.7	1.659	436.0	2.975	443.9
91	4.793	455.6	8.467	467.4	1.631	437.5	1.578	436.6	2.779	444.7
92	4.451	456.5	7.830	468.2	1.483	438.3	1.449	437.4	2.558	445.7
93	3.621	457.7	7.130	469.1	1.319	439.3	1.325	438.1	2.343	446.7
94	3.381	459.0	6.478	470.1	1.201	440.4	1.189	439.0	2.198	447.8
95	3.483	460.3	5.784	471.2	1.049	441.6	1.064	440.0	1.982	449.1
96	3.394	461.6	4.979	472.5	0.899	442.9	0.933	441.0	1.800	450.4
97	2.957	462.8	4.057	474.0	0.739	444.6	0.778	442.3	1.512	451.7
98	2.930	464.0	2.920	476.0	0.556	446.7	0.634	443.9	1.173	453.5
99	1.626	466.0	1.476	479.6	0.320	449.9	0.412	446.2	0.724	455.9

NYLON 6,10 100MG. SAMPLES
H.R. = HEATING RATE IN DEGREES C PER HOUR
RATE OF WT. LOSS IN PERCENT PER MINUTE

I.D./H.R.	06P/340		07P/520	
PERCENT WT.LOSS	RATE OF WT.LOSS	TEMP. DEG C	RATE OF WT.LOSS	TEMP. DEG C
1	0.251	350.2	0.891	362.1
2	0.	368.4	1.010	389.6
3	0.	389.5	1.071	399.5
4	1.484	398.4	1.634	406.6
5	0.924	403.9	2.407	411.4
6	2.467	407.3	3.068	414.0
7	2.152	409.9	3.641	417.1
8	3.175	412.0	4.740	419.0
9	3.313	414.1	3.380	421.3
10	3.354	415.6	4.570	423.1
11	3.779	417.2	6.297	424.7
12	4.489	418.5	7.215	425.9
13	4.939	419.7	8.352	427.0
14	5.313	420.9	8.492	428.0
15	6.017	422.0	8.422	429.0
16	6.377	422.8	8.986	430.0
17	6.490	423.7	9.990	431.0
18	6.730	424.5	11.030	431.8
19	6.385	425.4	11.937	432.6
20	6.701	426.4	12.235	433.3
21	7.187	427.2	12.427	434.0
22	7.463	428.0	13.239	434.6
23	7.662	428.7	13.470	435.3
24	8.191	429.5	13.870	435.9
25	8.697	430.1	14.401	436.5
26	8.796	430.8	14.783	437.1
27	9.026	431.4	15.387	437.7
28	9.401	432.0	14.571	438.2
29	9.751	432.6	14.814	438.8
30	9.616	433.1	15.583	439.4
31	9.673	433.7	15.020	439.9
32	9.874	434.3	15.328	440.5
33	10.215	434.9	15.819	441.1
34	10.464	435.4	15.661	441.6
35	10.860	436.0	16.186	442.2
36	10.897	436.5	16.469	442.7
37	11.115	437.0	16.796	443.2
38	11.293	437.5	17.374	443.7
39	11.238	438.0	17.891	444.2
40	11.455	438.5	18.255	444.6
41	11.643	439.0	17.843	445.1
42	11.952	439.5	18.469	445.6
43	12.031	440.0	17.553	446.0
44	12.086	440.4	17.838	446.5
45	12.242	440.9	16.660	447.1
46	12.328	441.3	16.011	447.6
47	12.720	441.8	16.174	448.1
48	12.708	442.2	16.358	448.7
49	12.764	442.6	17.189	449.2
50	12.887	443.1	18.883	449.7

PERCENT WT.LOSS	RATE OF WT.LOSS	TEMP. DEG C	RATE OF WT.LOSS	TEMP. DEG C
51	12.506	443.5	18.978	450.2
52	12.403	444.0	20.089	450.6
53	12.337	444.4	20.756	451.0
54	12.316	444.9	21.341	451.4
55	12.519	445.4	20.584	451.8
56	12.539	445.8	20.039	452.3
57	12.894	446.3	20.248	452.7
58	12.570	446.7	20.152	453.1
59	12.532	447.2	20.080	453.5
60	12.609	447.6	20.319	454.0
61	12.651	448.1	20.576	454.4
62	12.726	448.5	19.514	454.8
63	12.764	449.0	19.973	455.2
64	12.583	449.3	19.604	455.7
65	12.411	449.8	19.799	456.2
66	12.020	450.3	19.556	456.6
67	11.910	450.7	19.334	457.0
68	11.692	451.2	18.988	457.5
69	11.840	451.8	18.404	457.9
70	11.860	452.2	18.561	458.5
71	11.503	452.7	19.751	458.9
72	11.419	453.2	19.158	459.3
73	11.404	453.7	19.482	459.8
74	11.541	454.1	18.263	460.3
75	11.092	454.6	18.216	460.8
76	10.624	455.1	19.256	461.2
77	10.368	455.7	17.940	461.6
78	10.247	456.2	16.922	462.2
79	9.798	456.8	16.205	462.8
80	9.482	457.5	18.031	463.3
81	8.854	458.1	17.391	463.8
82	9.009	458.8	17.851	464.3
83	9.122	459.4	16.710	464.7
84	9.031	460.1	16.354	465.2
85	9.319	460.7	13.664	465.9
86	9.071	461.2	11.501	466.7
87	8.931	461.8	10.736	467.6
88	8.363	462.4	10.636	468.4
89	7.531	463.1	10.548	469.2
90	7.248	463.7	10.177	470.0
91	5.939	464.7	9.957	470.9
92	5.373	465.8	9.750	471.8
93	4.506	466.9	9.124	472.7
94	4.320	468.3	8.286	473.7
95	4.245	469.4	7.229	474.9
96	3.356	471.0	6.135	476.3
97	2.236	472.9	5.026	477.9
98	2.095	475.8	4.198	479.9
99	0.915	479.3	3.240	482.3

NYLON 6,6 100MG. SAMPLES
H.R. = HEATING RATE IN DEGREES C PER HOUR
RATE OF WT. LOSS IN PERCENT PER MINUTE

I.D./H.R.	01G/280		02G/450		03G/ 75		04G/150		05G/ 225	
PERCENT WT.LOSS	RATE OF WT.LOSS	TEMP. DEG C	RATE OF WT.LOSS	TEMP. DEG C	RATE OF WT.LOSS	TEMP. DEG C	RATE OF WT.LOSS	TEMP. DEG C	RATE OF WT.LOSS	TEMP. DEG C
1	0.231	339.2	0.354	252.6	0.040	330.2	0.107	326.8	0.033	296.7
2	0.370	364.0	0.279	339.4	0.120	346.4	0.242	347.9	0.073	328.7
3	0.691	372.5	0.	356.0	0.268	353.5	0.411	356.2	0.172	342.4
4	0.872	379.7	0.652	373.7	0.466	357.5	0.608	362.0	0.292	348.3
5	1.242	383.9	1.276	381.4	0.584	359.8	0.793	366.1	0.369	352.5
6	1.686	387.4	1.741	387.3	0.672	361.8	1.003	369.3	0.478	355.8
7	2.358	389.8	2.407	390.5	0.710	363.7	1.211	371.7	0.633	358.4
8	2.866	391.5	2.634	393.4	0.761	365.4	1.396	373.7	0.748	360.3
9	3.172	393.0	3.405	395.8	0.875	367.1	1.596	375.5	0.860	361.9
10	3.369	394.4	4.161	397.7	1.073	368.6	1.814	377.2	0.957	363.3
11	3.432	395.8	4.475	399.3	1.181	369.7	1.964	378.6	1.004	364.7
12	4.027	397.2	4.949	400.9	1.268	370.7	2.146	379.9	1.081	365.9
13	4.241	398.4	5.299	402.3	1.345	371.6	2.319	381.1	1.165	367.1
14	4.428	399.4	5.383	403.6	1.337	372.5	2.490	382.1	1.248	368.2
15	4.832	400.5	5.683	405.0	1.317	373.6	2.647	383.1	1.327	369.3
16	5.176	401.5	5.980	406.2	1.371	374.5	2.756	383.9	1.423	370.2
17	5.282	402.4	6.525	407.5	1.466	375.5	2.868	384.9	1.456	371.1
18	5.405	403.3	7.113	408.6	1.515	376.3	2.976	385.7	1.505	371.9
19	5.829	404.2	7.659	409.6	1.576	377.1	3.123	386.6	1.539	372.8
20	6.344	404.9	8.180	410.4	1.669	377.9	3.251	387.4	1.609	373.6
21	6.746	405.6	8.622	411.3	1.713	378.6	3.352	388.1	1.652	374.4
22	6.880	406.4	9.004	412.2	1.773	379.4	3.449	388.9	1.739	375.2
23	7.112	407.1	9.477	413.0	1.821	380.1	3.532	389.6	1.783	375.9
24	7.467	407.7	10.039	413.7	1.845	380.8	3.613	390.3	1.808	376.7
25	7.814	408.3	10.407	414.4	1.873	381.5	3.692	391.0	1.862	377.4
26	8.181	408.9	10.769	415.1	1.892	382.1	3.769	391.7	1.916	378.0
27	8.452	409.5	11.089	415.8	1.919	382.8	3.845	392.3	1.981	378.7
28	8.517	410.1	11.554	416.4	1.961	383.4	3.896	393.0	1.993	379.4
29	8.512	410.6	11.805	417.0	2.006	384.1	3.966	393.6	2.047	380.0
30	8.731	411.1	12.072	417.7	2.022	384.7	4.034	394.2	2.132	380.6
31	8.854	411.7	12.277	418.3	2.025	385.3	4.134	394.9	2.159	381.2
32	8.839	412.2	12.642	418.8	2.034	386.0	4.207	395.5	2.197	381.8
33	8.854	412.8	12.696	419.4	2.069	386.6	4.277	396.1	2.214	382.4
34	8.851	413.3	12.702	420.0	2.096	387.2	4.307	396.6	2.208	383.0
35	8.839	413.9	12.887	420.6	2.131	387.8	4.362	397.2	2.217	383.6
36	8.961	414.4	13.102	421.1	2.177	388.4	4.358	397.8	2.221	384.1
37	8.885	415.0	13.133	421.7	2.230	389.0	4.419	398.3	2.228	384.8
38	9.548	415.5	13.120	422.2	2.266	389.6	4.462	398.9	2.306	385.3
39	9.894	416.0	13.159	422.8	2.275	390.1	4.504	399.5	2.331	385.9
40	9.511	416.4	13.173	423.3	2.289	390.7	4.617	399.9	2.372	386.4
41	9.592	416.9	13.465	423.9	2.320	391.2	4.630	400.5	2.433	387.0
42	9.427	417.4	13.655	424.5	2.369	391.7	4.642	401.0	2.478	387.5
43	9.448	417.9	13.618	425.0	2.384	392.3	4.596	401.6	2.474	388.0
44	9.302	418.4	13.963	425.5	2.429	392.8	4.634	402.2	2.483	388.5
45	9.154	418.9	14.115	426.1	2.438	393.3	4.673	402.7	2.467	389.0
46	9.199	419.5	14.165	426.5	2.456	393.8	4.734	403.2	2.482	389.6
47	9.051	420.0	14.077	427.1	2.460	394.4	4.757	403.7	2.473	390.1
48	9.135	420.6	14.062	427.6	2.474	394.9	4.779	404.2	2.532	390.6
49	9.251	421.2	14.341	428.1	2.477	395.4	4.801	404.8	2.501	391.1
50	9.256	421.7	14.328	428.6	2.471	395.9	4.796	405.2	2.478	391.6

PERCENT WT.LOSS	RATE OF WT.LOSS	TEMP. DEG C	RATE OF WT.LOSS	TEMP. DEG C	RATE OF WT.LOSS	TEMP. DEG C	RATE OF WT.LOSS	TEMP. DEG C	RATE OF WT.LOSS	TEMP. DEG C
51	9.415	422.2	14.281	429.1	2.470	396.4	4.791	405.7	2.468	392.1
52	9.180	422.7	14.187	429.6	2.460	396.9	4.786	406.3	2.470	392.6
53	9.436	423.2	14.265	430.2	2.475	397.4	4.781	406.8	2.445	393.1
54	9.695	423.7	14.114	430.7	2.449	397.9	4.714	407.2	2.428	393.7
55	9.674	424.2	14.206	431.2	2.437	398.4	4.688	407.8	2.421	394.2
56	9.652	424.7	14.026	431.7	2.412	399.0	4.657	408.3	2.453	394.8
57	9.685	425.2	13.933	432.2	2.380	399.5	4.619	408.8	2.498	395.3
58	9.660	425.7	14.000	432.8	2.371	400.0	4.581	409.4	2.532	395.8
59	9.692	426.2	14.061	433.3	2.382	400.5	4.605	409.8	2.522	396.3
60	9.724	426.7	14.057	433.9	2.388	401.1	4.549	410.4	2.523	396.8
61	9.868	427.2	14.053	434.4	2.374	401.6	4.491	410.9	2.523	397.3
62	9.922	427.7	14.011	434.9	2.353	402.1	4.433	411.5	2.468	397.8
63	9.831	428.1	13.684	435.4	2.331	402.7	4.441	412.1	2.452	398.3
64	9.621	428.6	13.607	436.0	2.310	403.2	4.385	412.6	2.414	398.8
65	9.708	429.1	13.517	436.5	2.288	403.8	4.328	413.2	2.389	399.4
66	9.686	429.6	13.437	437.0	2.265	404.3	4.271	413.8	2.350	399.9
67	9.594	430.1	13.415	437.6	2.243	404.9	4.187	414.3	2.326	400.4
68	9.677	430.5	13.316	438.2	2.227	405.4	4.122	414.9	2.305	401.0
69	9.508	431.0	13.400	438.7	2.205	406.0	4.060	415.5	2.219	401.6
70	9.409	431.6	13.559	439.2	2.136	406.6	3.994	416.2	2.218	402.1
71	9.390	432.0	13.275	439.8	2.126	407.2	3.927	416.8	2.113	402.7
72	9.235	432.6	12.984	440.3	2.106	407.8	3.858	417.4	2.069	403.3
73	9.077	433.1	12.721	440.9	2.079	408.4	3.753	418.1	2.049	404.0
74	8.841	433.6	12.471	441.5	2.039	409.0	3.691	418.8	2.027	404.6
75	8.540	434.1	12.488	442.1	1.994	409.6	3.628	419.4	1.992	405.2
76	8.303	434.7	12.193	442.7	1.934	410.2	3.553	420.1	2.011	405.9
77	8.363	435.3	12.038	443.3	1.866	410.9	3.484	420.8	1.939	406.5
78	8.269	435.9	11.901	444.0	1.790	411.6	3.414	421.5	1.897	407.1
79	8.120	436.4	11.500	444.5	1.725	412.3	3.343	422.2	1.808	407.8
80	7.913	437.0	11.136	445.2	1.652	413.1	3.279	422.9	1.698	408.5
81	7.435	437.6	10.941	445.9	1.593	413.8	3.191	423.6	1.605	409.3
82	7.258	438.3	10.604	446.5	1.502	414.7	3.100	424.4	1.569	410.2
83	7.046	439.0	10.237	447.2	1.434	415.5	3.007	425.2	1.533	410.9
84	6.744	439.6	9.773	448.0	1.341	416.4	2.933	426.0	1.457	411.8
85	6.499	440.3	9.443	448.8	1.258	417.4	2.828	426.8	1.399	412.7
86	6.226	441.1	8.861	449.6	1.229	418.4	2.683	427.7	1.303	413.6
87	5.594	442.0	8.413	450.4	1.132	419.5	2.554	428.6	1.188	414.7
88	5.366	442.8	8.029	451.3	1.103	420.6	2.444	429.6	1.191	415.7
89	5.147	443.6	7.625	452.2	1.016	421.7	2.310	430.6	1.159	416.8
90	4.844	444.6	7.198	453.2	0.854	423.2	2.163	431.8	1.137	417.8
91	4.434	445.6	6.745	454.3	0.865	424.6	2.031	432.9	0.998	419.1
92	3.978	446.7	6.259	455.4	0.892	425.9	1.936	434.1	0.880	420.4
93	3.349	448.0	5.883	456.6	0.807	427.4	1.754	435.4	0.792	421.9
94	2.951	449.6	5.417	457.9	0.711	429.1	1.578	436.9	0.707	423.6
95	2.579	451.3	4.726	459.4	0.597	431.1	1.438	438.5	0.646	425.5
96	2.184	453.1	4.414	461.1	0.487	433.3	1.273	440.3	0.584	427.6
97	1.766	455.4	3.607	462.9	0.361	436.3	1.101	442.2	0.504	430.0
98	1.377	458.2	2.791	465.7	0.280	440.2	0.928	444.3	0.367	432.9
99	0.814	462.5	1.739	469.0	0.013	447.3	0.587	447.5	0.206	437.9

UNCLASSIFIED

Security Classification

DOCUMENT CONTROL DATA - R & D

(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)

1. ORIGINATING ACTIVITY *(Corporate author)*

Air Force Materials Laboratory
Wright-Patterson Air Force Base, Ohio 45433

2a. REPORT SECURITY CLASSIFICATION

Unclassified

2b. GROUP

3. REPORT TITLE

THERMAL DEGRADATION OF POLYAMIDES
PART I. ALIPHATIC POLYMERS

4. DESCRIPTIVE NOTES *(Type of report and inclusive dates)*

September 1966 to June 1968

5. AUTHOR(S) *(First name, middle initial, last name)*

Goldfarb, Ivan J.
Meeks, A. C.

6. REPORT DATE	7a. TOTAL NO. OF PAGES	7b. NO. OF REFS
January 1969	94 15	37

8a. CONTRACT OR GRANT NO.	9a. ORIGINATOR'S REPORT NUMBER(S)
b. PROJECT NO. 7342	AFML-TR-68-347, Part I
c. Task No. 734203	9b. OTHER REPORT NO(S) *(Any other numbers that may be assigned this report)*
d.	

10. DISTRIBUTION STATEMENT

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11. SUPPLEMENTARY NOTES	12. SPONSORING MILITARY ACTIVITY
	Air Force Materials Laboratory Wright-Patterson Air Force Base, Ohio 45433

13. ABSTRACT

The thermal degradation of two aliphatic polyamides, polyhexamethylene adipamide (nylon 6.6) and polyhexamethylene sebacamide (nylon 6.10) have been studied. Molecular weight changes, weight loss, and volatile product analysis were used to help elucidate the reaction mechanisms.

The presence of low molecular weight material and polymerizable end groups in these polymers complicated the interpretation of molecular weight changes during degradation. The weight loss data obtained allowed the calculation of rate data. Nylon 6.6 degradation gave an activation energy of 45 kcal/mole while nylon 6.10 degradation was characterized by an activation energy of 55 kcal/mole. Both polymers gave evidence of random scission kinetics. The volatile products were consistent with the occurrence of further condensation, scission, and cross-linking reactions.

DD FORM 1 NOV 65 1473

UNCLASSIFIED

Security Classification

14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Thermal Degradation						
Polymer						
Polyamide						
Nylon						